ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL, FISCAL YEAR 1950

BY ARNO C. FIELDNER AND SIDNEY GOTTLEY

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Arno C. Fieldner[1] and Sidney Gottley[2]

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INTRODUCTION

From July 1, 1949, to July 1, 1950, the Bureau of Mines conducted the research and technologic work on coal and coal products that is summarized in this report. The research, described in this fifteenth of a series of résumés, is presented in more detail in publications (see footnote references) that appeared during the fiscal year, though in some cases research data are given that have not been published elsewhere. Results of research in the experimental coal mine, on explosives, and on gas and dust explosions will be found in Bureau of Mines Report of Investigations 4228, Annual Report of Research and Technologic Work on Explosives, Explosions, and Flames, Fiscal Year 1950.

An over-all view of our coal, oil, and natural-gas reserves was presented, and the future possibilities for the production of synthetic liquid fuels from coal were delineated.[3]

A study of future petroleum supply and demand[4] indicated that by 1975 the bulk of our oil may have to come either from foreign countries or from alternate domestic sources such as coal. Coal, the most abundant alternate source of liquid fuels, can be converted to gasoline and oils by two processes. Quantities and characteristics of coal requisite for each process were discussed, and cost data for products were presented. The current status of the Bureau of Mines demonstration plants for the production of synthetic liquid fuels from coal and new developments in processes for such production were reported.[5][6]

Current and future aspects of chemistry in the coal industry were considered,[7] with a brief résumé of the present situation in coal reserves and production.

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[3] Fieldner, A. C., The National Fuel Reserves and Future Fuel Supplies: Coal Mining Modernization Handbook, 1949, pp. 374-390.

[4] Doherty, J. D., Synthetic Oil from Coal: Colorado School of Mines Quarterly, vol. 45, No. 2B, April 1950, pp. 77-95; discussion pp. 95-105.

[5] Schroeder, W. C., Synthetic Liquid Fuel - Two Coal-to-Oil Demonstration Plants Are Producing Liquid Fuels from Coal on the Minimum Scale Enabling the Bureau of Mines to Make Available to Industry the Necessary Cost and Engineering Data for Commercial Operations: Armed Forces Chem. Jour., vol. III, No. 5, July 1949, pp. 12-13.

[6] Schroeder, W. C., Synthetic Fuel Production: Petrol. Eng., vol. 22, May 1950, pp. A-53, 56, 60, 63, 64, 66, 68.

[7] Hunter, T. W., Mineral Fuels (in Basic Minerals - Production High But Reserves Cause Concern): Ind. Eng. Chem., vol. 42, June 1950, pp. 978-983.

Progress in coal-mine safety research from 1910 to 1950 was reviewed.[8]

Construction of the first wing of the Anthracite Research Laboratory at Schuylkill Haven, Pa., was completed, and research programs were initiated, while construction of the Lignite Research Laboratory at Grand Forks, N. Dak., was well under way.

SUMMARY

While total coal supplies were sufficient for the country's needs, certain areas lacked adequate supplies of solid fuels and their products for special purposes. The Bureau of Mines research program on the mining, preparation, and utilization of coal was prosecuted vigorously to insure development of reserves and supplies of such coals, without sacrifice of safety, efficiency, and conservation.

To clarify our reserve position and augment our dwindling reserves of coking coal, the Bureau continued its estimation of known minable reserves of such coals in central and southern Pennsylvania, southern West Virginia, and eastern Kentucky. This survey was supplemented by field investigations in the Georges Creek Basin in Maryland, where diamond drilling revealed estimated reserves of coking coal in excess of 399,000,000 tons, of which 50 to 60 percent is recoverable. In the laboratory, the problems of alleviating shortages of coal suitable for metallurgical coke were attacked by studies of the washing and blending characteristics of lower-grade coals, not now usable for coke making. These studies provided essential data on the carbonizing properties of 46 American coals and of blends of these coals to guide coke producers in selecting proper coals for their ovens. Research on the low-temperature carbonization of lower-rank coals was initiated, and a survey of the yields and properties of the products of such carbonization from representative western coals was made. Determination of the preparation characteristics of coals from Illinois, Tennessee, Pennsylvania, and West Virginia demonstrated the feasibility of upgrading many of these coals by preparation methods. Research on the desulfurizing of high-sulfur coals gave information on the possibilities of using ammonia gas for this purpose.

Conservation of our coal resources through efficient utilization was the goal of much of the Bureau's program. As in previous years, the Bureau of Mines acted as a consultant to other Federal agencies on the purchase of fuel, fuel-burning and boiler-room equipment, and on boiler-water chemical treatment. Analyses and recommendations were made on 9,335 samples of boiler water, and 580 Bureau of Mines field water-test kits, 10,259 bottles of chemical reagents, and 12,060 test-kit-replacement items were distributed to various Government activities. Proximate or ultimate analyses were made on 10,100 samples from purchases of coal and tipple and breaker inspections on which Federal fuel purchases were based. The savings resulting from these consulting services continued at a level many times their cost, and boiler-water research indicated even greater savings might be forthcoming in the future. Studies of the mechanism of external corrosion of furnace-wall tubes in boiler furnaces provided information that can greatly reduce the cost of equipment replacement and out-of-service time. In cooperation with the American Institute of Mechanical Engineers, the Bureau made further determinations of furnace-heat absorption in utility power boiler furnaces, which help to improve efficiency of operation, with consequent increases in fuel conservation.

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[8] Fieldner, Arno C., Achievements in Mine Safety Research and Problems Yet to be Solved: Bureau of Mines Inf. Circ. 7573, 1950, 31 pp.

Continued emphasis was placed on the development of feasible methods of utilizing our vast reserves of low-rank coals in the West, whose high natural-moisture content places them at a disadvantage in competition with higher-rank coals. Bureau investigations of drying these coals in the entrained and fluidized state promised to offset this disadvantage.

A new technique, involving the use of replica films, was developed for utilizing the electron microscope in studying the microstructure of coal. Shortages of montan wax for industrial use led to a survey of the waxes extractable from American lignites, with Arkansas and California lignites providing the highest yields of wax in solvent extraction tests.

Seeking to increase recovery and efficiency to meet primary problems of conservation in the coal industry, the Bureau studied the extraction of pillars underground with mechanized equipment and demonstrated that such extraction increases percentage recovery of coal, while decreasing production per mobile loading machine. In the anthracite fields of Pennsylvania, mechanization was advanced by successful underground tests on German shearing and packing machines and on the Bureau-developed scraper-shaker loader. Research was initiated on new methods of roof support in anthracite mines that will greatly reduce injuries to miners from roof falls. Another major problem in coal conservation was the huge quantities of coal consumed by uncontrolled mine fires. Sealing, flooding, and carbon dioxide were employed to control such fires.

Striving to achieve greater safety in mining coal, Bureau research yielded data on the effect of operating conditions on the composition of exhaust gases from Diesel engines used underground that is essential to the solution of many operating and combustion problems. To overcome harmful atmospheric conditions in coal mines, several studies were completed on the determination, collection, and dissemination of air-borne dusts and on methods of their control. More than 16,000 samples of mine air were analyzed, and over 1,000 samples of air-borne dusts were examined to determine the adequacy of ventilation in coal mines, as well as to detect and aid in the elimination of hazards from gases and mine fires. Eight new approvals and 25 extensions of approvals were granted for respiratory protective devices tested by the Bureau.

Research on the gasification of coal and lignite was continued, including the second underground gasification project at Gorgas, Ala. In this experiment, the effect of various conditions of flow on the heating value of the gas produced has been determined. Experimentation with the atmospheric-pressure pilot plant to produce synthesis gas by the gasification of pulverized fuel reduced oxygen requirements for the gasification of coal to the lowest yet reported for any continuous process; a new unit was developed that permitted the testing of various types of coal for their utility in making synthesis gas on a commercial scale. An important phase of the gasification research was the purification of the synthesis gas for use in synthesizing liquid fuels. Laboratory and pilot-plant hydrogenation of coal to produce liquid fuels was studied in both liquid- and vapor-phase units. Greatly improved yields of soluble materials from coal resulted from using dicyclic hydroaromatic compounds, containing at least one phenolic hydroxyl group, as new solvent vehicles. Research to discover new, more readily available catalysts, as reactive as tin in coal hydrogenation, was particularly comprehensive. The factors involved in cleaning coal for hydrogenation to eliminate oil loss due to the presence of ash were studied.

At Louisiana, Mo., the Coal-Hydrogenation Demonstration Plant was completed and went through the necessary break-in procedures. The Gas-Synthesis Demonstration Plant was practically complete, and several of the units within the plant were successfully operated. As these processes demonstrated their technical feasibility, much interest was shown in the economic aspects of their application. Many estimates were made to determine the commercially competitive position of liquid fuels derived from coal.

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ORIGIN, COMPOSITION, AND PROPERTIES OF COAL

Inspection, Sampling, and Analysis

At the beginning of the fiscal year 1950, coal was being produced at a rate equal to or in excess of demand, and Federal agencies had no difficulty in obtaining bids to cover their requirements. Owing to the shift from a "seller's" to a buyer's" market, all agencies were able to make their purchases on a guaranteed-analysis basis, including penalties for delivery of substandard coal. This method had been largely abandoned during the coal shortage of previous years. The Navy Department, purchasing coal for the Army, Air Force, and Navy, was the largest Federal coal-purchasing agency. In general, other departments purchase their own coal supply, except that the Federal Supply Service purchases the supply for some other agencies whose requirements are small. Contracting for coal for the fiscal year 1951 started in March and continued through the balance of the fiscal year.

Coal requirements of Federal agencies for the fiscal year were estimated to be approximately 5,000,000 tons. There was a large increase in requests from Federal agencies for analyses to be used in evaluating coal bids. This was caused in part by an increase in the number of bids received and in part by a more strict adherence to specification requirements that eliminated mines on which there were no Bureau of Mines analysis records.

At the urgent request of Federal agencies, mine operators, and coal-sales agencies for more service in obtaining mine-tipple analyses, the sampling program was expanded. A total of 1,505 tipple samples was collected at 481 mines in 11 States. At the beginning of the fiscal year, requests for sampling more than 600 mines were on file, with additional requests received daily. At the close of the year, about 350 mines still remained unsampled. For carbonization, grindability, electrode carbon, and other special tests, 447 samples were collected in 441 mines. At the same time, 36,000 tons of coal exported to Japan were inspected and sampled at Norfolk, Va.

To promote more accurate sampling of delivered coal and reduce sampling costs, many Government installations have installed mechanical equipment for preparing samples. It became necessary to issue new instructions[9] for coal sampling to cover this innovation. At the request of the Navy Department and Marine Corps, special instructions were given in coal sampling at five installations.

Data in the first of the Bureau's series of technical papers describing coals of individual producing States, Technical Paper 269, Analyses of Iowa Coals, has been brought up to date by analyses made since its publication in 1921, and the revised and expanded material was published.[10] The geology of the coal fields, methods of mining, preparation, production, distribution, uses, and the relationship of mine samples to commercial shipments were discussed. Descriptions of mine, tipple, and delivered samples were given, and chemical analyses, calorific values, classifications by rank, agglomerating indexes, and fusing temperatures of ash were included. The locations of operating mines were included, with data on the nature of roof and floor and on thickness of coal and partings in the beds.

Proximate or ultimate analyses were reported on 10,100 samples from purchases of coal and tipple and breaker inspections taken in connection with Federal fuel purchases, an increase of 47 percent over the previous year. Fusing-temperature determinations were reported on 2,980 of the samples.

From 1,713 inspections of coal mines by the Bureau's coal-mine inspectors, a total of 7,984 samples of road, roof, rib, and gob dust was received and analyzed. These samples, usually received, analyzed, and reported on the same day, guide the inspectors in determining if enough rock dusting is being done in mines to insure safe operation.

The total of 24,681 samples received was not only the largest number ever handled by the laboratory, exceeding the previous year's record number by 565, or 2.3 percent, but, in addition, the actual analytical work involved was even greater because of a large increase in the number of determinations per sample, since more ultimate and proximate analyses were required by more rigid standards in coal purchasing and in connection with foreign cooperative projects; the work-load increase was 9.0 percent.

The averages and the ranges of as-received B.t.u. values of American coals on a national and also on a State basis were determined.[11] Table 1 gives the national averages:

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[9] Snyder, N. H., Handbook on Coal Sampling: Bureau of Mines Handbook, 1950, 10 pp. (Revision of Tech. Paper 133, Directions for Sampling Coal for Shipment or Delivery, by G. S. Pope, revised by N. H. Snyder.)

[10] Averitt, Paul, Toenges, Albert L., Olin, H. L., Bell, C. H., Snyder, N. H., Cooper, H. M., Abernethy, R. F., and Tarpley, E. C., Analyses of Iowa Coals: Tech. Paper 706, 1949, 65 pp.

[11] Flynn, George J., Jr., Average Heating Values of American Coals by Rank and by States: Bureau of Mines Inf. Circ. 7538, 1949, 11 pp.

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TABLE 1. - Average B.t.u. values of American coals

Rank	As-received B.t.u. content, average
Anthracite (Pennsylvania only)	12,750
Bituminous (all)	13,100
Low-volatile bituminous	14,200
Medium-volatile bituminous	14,000
High-volatile bituminous	12,900
Subbituminous	9,550
Lignite	7,000
All ranks	13,000

A chapter covering sampling, analysis, and properties of coal and other solid fuels was prepared for the twelfth edition of Kent's Mechanical Engineers' Handbook.[12] Methods of sampling coal and coke, methods of preparing and analyzing laboratory samples, properties of coal and of coal ash, coal specifications, bulk density and miscellaneous related data, and typical analyses of coals from large mines actively in production in 1948-49 were described. Properties and analyses of coke, wood, hogged fuels, and miscellaneous solid fuels were also included.

A number of samples of coals from the United States and from European countries were analyzed in connection with the International Coal Classification Program, wherein there was an interchange of samples among the national coal laboratories of most of the coal-producing countries as part of a program to develop an international standard coal-classification system. Cooperation was extended to the Economic Cooperation Administration through analysis of Greek and Korean coals.

Analyses of Miscellaneous Materials

To determine whether certain dusts were suitable for rock dusting in coal mines to prevent coal-dust explosions, 10 samples of limestone, marl, boiler fly ash, etc., were chemically analyzed; 3 were found satisfactory as submitted, and 2 would be if ground finer. A proprietary fuel-saver and 11 other miscellaneous compounds were analyzed to determine their chemical nature. The chemical constituents of 75 coal ash, coal-ash slag, and boiler-water and tube-deposit samples were determined to aid the Bureau's research programs and assist in making recommendations for better operation of boilers and better feed-water treatment.

Constitution, Properties, and Analytical Methods

Coal Classification

A review and discussion of coal classification by rank, grade, and type was published.[13]

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[12] Carman, E. P., Corey, R. C., Morgan, R. E., and Brewer, R. E., Solid Fuels: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-17 to 2-45, John Wiley & Sons, Inc., New York, N. Y.

[13] Carman, E. P., Solid Fuels. 6. Coal Classification: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-17 to 2-20, John Wiley & Sons, Inc., New York, N. Y.

Petrographic Studies of Coal

The Georges Creek and Upper Potomac coal beds, Allegany and Garrett Counties, Md., were investigated by diamond-drill-hole testing and laboratory study of recovered cores.[14] All cores from the test holes were studied megascopically and selectively sampled for special analyses and tests. Cores of six commercially important beds studied revealed bright, banded coals having numerous thin lenses of mineralized fusain and pyritic impurities. Thin sheets of mineral impurities, such as calcite and kaolinite, were common to the vertical fracture surfaces. Figure 1 illustrates graphically the character of typical sections of the commercially important coal beds - Barton, Harlem, Lower Freeport, Upper Kittanning, Middle Kittanning, and Lower Kittanning.

These observations indicate that much of the ash-forming impurities in certain coal beds consist of mineral matter associated with the coal in form of layers, lenses, and veinlets. The extraneous mineral impurities, which are of much higher specific gravity than the coal, can be removed from the coal by gravity-separation methods of coal cleaning, thereby improving the quality of the coal for combustion and coking uses.

Electron Microscopy of Coal

A technique has been developed for utilizing the electron microscope in studies of coal microstructures.[15] The high resolving power of this instrument will be of value in studies of variations in coal microfossils that are found in different beds and areas. These differences in species are used as a basis for identification and correlation of different coal beds in surveys of national coal resources.

The usual thin-section or "peel" methods of coal petrography are inadequate for electron microscopy. In the new technique, the coal specimens are polished by the usual methods, then etched for a short period in a chromic acid-sulfuric acid etchant. After washing and drying, a double film of Formvar (polyvinyl formal)-nitrocellulose is formed on the surface by successively applying solutions of these resins. The film is stripped from the coal surface, specimen screens are fastened over selected areas, and the nitrocellulose is dissolved in amyl acetate. The remaining Formvar film (about 0.1 micron thick), in which the contours and fine details of the specimen surface are reproduced, is observed in the electron microscope.

Figure 2, a sample electron micrograph of coal structures reproduced in such a replica, shows a replica of attrital coal containing microspores, which appear lighter in the micrograph. Many of the details of compression and folding shown here would not be shown clearly by the optical microscope.

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[14] Toenges, Albert L., Turnbull, Louis A., Williams, Lloyd, Smith, H. L., O'Donnell, H. J., Cooper, H. M., Abernethy, R. F., and Waage, Karl M., Investigation of Lower Coal Beds in Georges Creek and North Part of Upper Potomac Basins, Allegany and Garrett Counties, Md.: Reserves, Petrographic and Chemical Characteristics of Coals, and Stratigraphy of Area: Bureau of Mines Tech. Paper 725, 1949, 142 pp.

[15] McCartney, J. T., Electron Microscopy of Coal: Econ. Geol., vol. 44, November 1949, pp. 617-620.

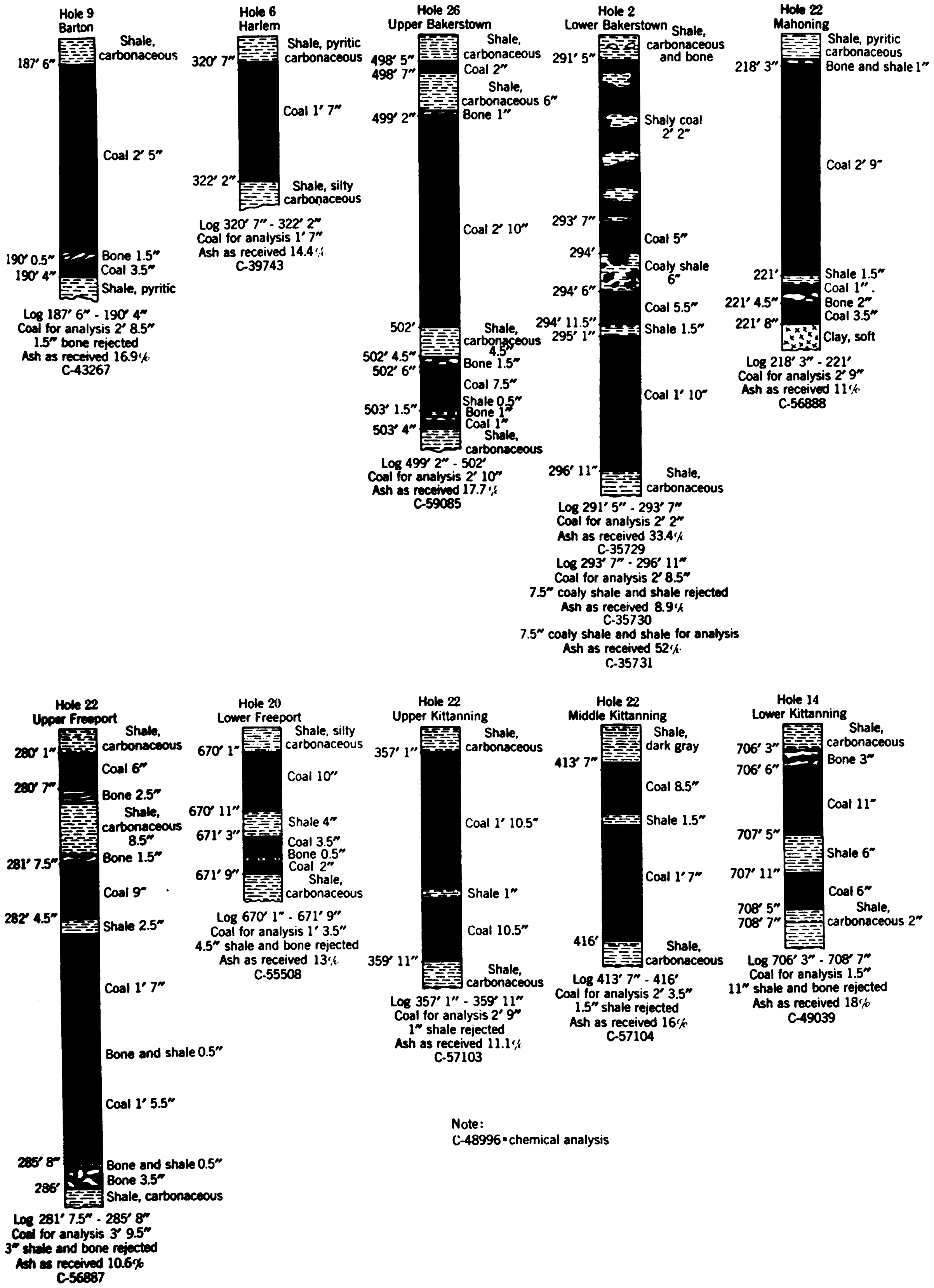

Figure 1. - Typical coal sections of drill cores from Georges Creek and north part of Upper Potomac Basins, Allegany and Garrett Counties, Md.

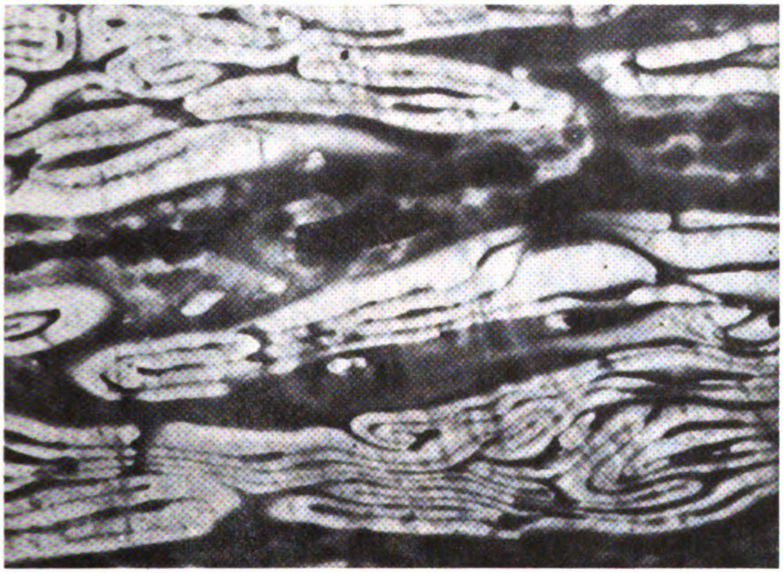

Figure 2 - Electron micrograph of a Formvar replica of microspores in attrital coal. (X4,500.)

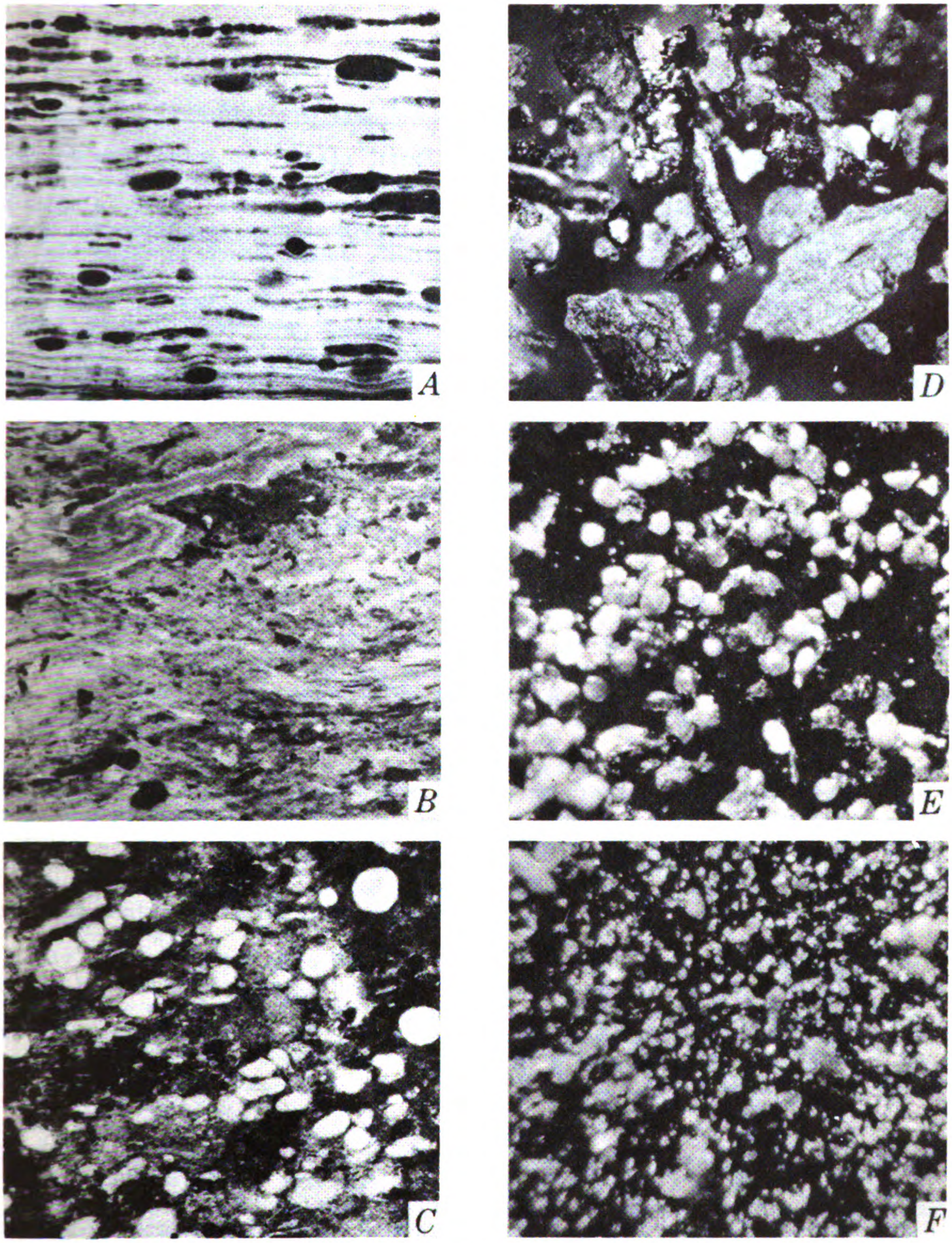

Figure 3. - Photomicrographs of thin sections of lignitic coal and ingredients isolated from attrital lignite; A, Coal material derived from woody tissues called anthraxylon and the chief component of xyloid lignite (X200); B, Orange-colored humic material, an important component in xyloid and some attrital lignites (X 100); C, Attrital type lignite containing large, yellow translucent particles (X 100); D, Resin particles and cuticular material isolated from attrital lignite, plus 180-mesh (X40); E, Resin particles of round shape isolated from attrital lignite, 180- x 325-mesh (X67); F, Spores, pollen, and fine, yellow granular material from attrital lignite, minus 325-mesh (X 100).

Extractable Waxes from American Lignites

Solvent extraction of certain lignitic coals yields a wax termed "montan wax." The commercial grade of montan wax is a high-melting, hard, brittle wax which has many industrial uses. It is an important ingredient in certain polishes, electrical insulating compositions, leather dressings, inks, carbon papers, protective coatings, greases, and other products. Montan-wax requirements for industry in the United States before World War II were met by imports, nearly all of which came from Germany. Since the war, montan wax has not been available from Germany, but relatively small amounts have been imported from Czechoslovakia.

Because of the shortage of montan wax at the close of the war and the fact that American industry in the past had to rely on foreign sources, a laboratory investigation was started in 1945 to obtain information concerning yields and properties of solvent-extractable wax from domestic lignites. Published results of the investigation[16/][17/] include discussions of the occurrence and geology of lignite deposits of the United States, from which samples were obtained for wax extraction, and the petrographic composition of lignites, particularly in respect to those components that contribute to wax yields.

The survey of the solvent-extraction yields showed that the highest yields were obtained from certain Arkansas and California lignites. Some of these yields, particularly those obtained with a mixture of benzene and alcohol, were similar to those obtained in commercial extraction of montan wax from German brown coal. The lignites examined from Montana, North Dakota, Texas, and Washington gave appreciably smaller quantities of solvent extract than the Arkansas and California lignites.

Although higher yields of extract were obtained with a solvent mixture of benzene and alcohol than with benzene alone, the properties of the benzene extracts more closely resembled commercial grades of imported montan wax than did the benzene-alcohol extracts. The most significant difference between the extracts obtained in this investigation and the Riebeck brand of montan wax from Germany was the greater resin content of the extracts from domestic lignites; they more closely resembled a commercial grade of montan wax from Czechoslovakia.

A study of the petrographic composition of American lignites showed that the high wax-yielding lignites are attrital and consist predominately of plant remains of small particle size, called attritus, much of which is yellow translucent matter under the microscope (fig. 3, C). Woody or xyloid lignites contain only small quantities of extractable wax; they consist largely of orange, translucent coal material of relatively large particle size, which was derived mainly from wood tissues of plants (fig. 3, A and B).

The main source of the soluble material extracted from lignite, which consists of wax, resin, and asphaltic material, was certain ingredients in the attrital ground mass. These ingredients were yellow cuticular material, round resinous bodies, lump-type resinous particles, spores, pollen, and very fine, yellow granular matter of uncertain origin (fig. 3, D, E, and F). Cuticular material was undoubtedly the original source of much of the waxy fraction.

16/ Selvig, W. A., Ode, W. H., Parks, B. C., and O'Donnell, H. J., American Lignites: Geological Occurrence, Petrographic Composition, and Extractable Waxes: Bureau of Mines Bull. 482, 1950, 63 pp.

17/ Ode, W. H., and Selvig, W. A., Extractable Waxes from American Lignites: Ind. Eng. Chem., vol. 42, January 1950, pp. 131-135.

COAL MINING

Coal Investigations

Alaska

The results of an investigation of coal mining in the Matanuska and the Nenana fields of Alaska were reported.[18] Alaska requires an adequate fuel supply for its development and has large potential coal reserves ranging from lignite to bituminous, with a little anthracite, but coal production in the Territory is now less than requirements. In 1947 production was 361,000 tons, divided about equally between bituminous coal from the Matanuska field and subbituminous coal from the Nenana field.

The coal exposures in the Matanuska field occur in an area approximately 25 miles long by 7 miles wide and parallel the general syncline of the Matanuska Valley. The coal beds, which are predominantly bituminous in rank, occur in the Chickaloon formation of Tertiary age. At present the Evan Jones mine is the only mine in operation in this field. Other mines have been worked in the past, but physical conditions in and surrounding the beds have been a factor in closing the mines. Beds of low-rank subbituminous coal and lignite occur in the Nenana field, which is characterized by the absence of extreme structural disturbances and igneous intrusions. Coal has been produced from one underground mine, the Suntrana mine of the Healy River Coal Corp., and from the Usibelli and the Diamond strip mines.

Greek Lignite

Following investigation of Greek lignite deposits by a Bureau of Mines engineer, recommendations were made and plans were submitted to the Economic Cooperation Administration for a diamond-drilling program at Kimi and Aliveri on the Island of Euboea. The objective of this program was to determine the minable reserves of lignite in these areas that could be developed to provide fuel for power generation and for chemical uses. The two areas were diamond-drilled under the supervision of a Bureau of Mines engineer. From September 1949 to March 8, 1950, 1,980 feet in two diamond-drill holes were drilled at Aliveri, and seven holes, totaling 5,035 feet, were drilled at Kimi.

Georges Creek, Md.

The results of an investigation of the lower coal beds in Georges Creek and north part of the Upper Potomac Basins, Allegany and Garrett Counties, Md., were published.[18a]

The annual production of coal from mines in the Georges Creek field in Allegany and Garrett Counties, Md., has declined from 2,250,000 tons before 1931 to about 1,800,000 tons in the past few years. The greater part of this production was from mines being worked in the upper coal beds - Pittsburgh [Big Vein and Sewickley (Tyson)] - which are approaching depletion after many years of mining. Because of this depletion, the annual production of the field is certain to decline further, with an accompanying loss of employment for miners, unless production from the lower, thinner beds can be increased.

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[18] Toenges, A. L., Coal-Mine Development in Alaska: Am. Inst. Min. and Met. Eng. Tech. Pub. 2686F, Min. Trans., vol. 184, 1949, pp. 361-364; Min. Eng., vol.1, October 1949, pp. 361-364.

[18a] See footnote 14.

The coals of the lower beds are low-volatile, strongly coking coals, some of which are suitable for blending in the manufacture of metallurgical coke. Another important use for this semismokeless coal is for domestic and industrial use in the nearby Washington, Baltimore, and Pittsburgh areas. Little was known of the continuity and thickness of these lower beds; the purpose of this investigation was to determine the reserves, thickness, physical characteristics, and chemical properties of these coals, with the objective of obtaining necessary data for developing mines in the lower beds.

The area was investigated by diamond drilling, engineering examination, and geologic study. Operating and abandoned mines, where accessible, were examined, and all available maps and information regarding mined-out areas were obtained. Thirteen beds of coal were penetrated in drilling. These beds, from the top downward are: Barton, Federal Hill, Harlem, Upper Bakerstown, Lower Bakerstown, Brush Creek, Mahoning, Upper Freeport, Lower Freeport, Upper Kittanning, Middle Kittanning, Lower Kittanning, and Mount Savage. The total estimated reserves of coal (including measured, indicated, and inferred reserves), 18 inches or more thick in 10 beds, are as follows:

Coal bed:	Estimated reserves, tons
Barton	63,318,000
Harlem	16,651,000
Upper Bakerstown	79,435,000
Lower Bakerstown	131,393,000
Mahoning	18,432,000
Upper Freeport	146,926,000
Lower Freeport	3,042,000
Upper Kittanning	94,902,000
Middle Kittanning	61,907,000
Lower Kittanning	10,723,000
Total	626,729,000

Possible reserves in the Federal Hill, Brush Creek, and Mount Savage beds were not estimated, because these beds are either too thin or not extensive enough to be important at present.

The minimum thickness of bed used in estimating reserves is 18 inches, but it may not be economical to mine a bed of this thickness in this field at present. The minimum thickness that can be mined depends upon many economic factors.

The bed moisture ranges from 1.5 to 3.0 percent, and the dry, ash-free volatile matter for all of the coals averages 21 percent. The ash and sulfur contents of the coals is relatively high and varies considerably among the different beds and within the same bed in the same area. The weighted average ash analysis of all samples analyzed on the moisture-free basis (excluding samples of the Mount Savage coal) ranges from 12.3 percent for the Federal Hill bed to 21.2 percent for the Upper Bakerstown bed; the weighted average sulfur, on the same basis (excluding the Mount Savage coal), ranges from 1.8 percent for the Mahoning bed to 4.5 percent for the Lower Kittanning bed. However, the ash and sulfur contents of these coals can be improved by washing.

The correlation of the coal beds penetrated in drilling was made possible by a study of the stratigraphy of the Georges Creek and the north part of the Upper Potomac Basins. The principal control in hole-to-hole correlation was the persistent sequence of key beds in the lower part of the Conemaugh formation between the Barton and the Brush Creek coals. Below the Brush Creek coal, the intervals between coal beds were the only possible control because key beds, other than coals, were not present. The names used for coal beds, other than the Mount Savage coal, follow Pennsylvania terminology, which is standard for the Northern Appalachian coal field.

Study of Coking-Coal Reserves

In the study of coking-coal reserves, field investigations in areas in central and southern Pennsylvania, southern West Virginia, and eastern Kentucky were completed. Estimates of reserves in these areas are in preparation. A report of the estimated known recoverable reserves of coking coal in Cambria County, Pa., the first in a series of reports by counties, was completed, and a report on Indiana County, Pa., reserves is almost complete.

Mining Methods and Practices

Bull Mountain, Mont.

In the course of studies of coal-mining methods and practices, electrical power requirements at coal mines, and coal resources in the Missouri River Basin, the results of an investigation in the Bull Mountain coal field of Montana were published.[19] This report contains a short summary of the geology and geography of the field and discusses the following information obtained during the field study by Bureau of Mines engineers: Method of mining practiced at each mine, including a description of each phase of the mining cycle; coal produced and its distribution at each mine; power requirements and the consumption of electricity per ton of coal produced by mines; number of men employed by mines, the production per man per shift underground, and the over-all production per man employed; and estimates of future production and power requirements for the field as a whole.

Iowa

A description of methods of mining in Iowa coal mines was published.[19a] Iowa ranks eighteenth in the United States in the production of bituminous coal and lignite, with output of 1,670,156 tons in 1948. The greater part of this production was from mines in the central and south central parts of the State. Physical conditions vary in these areas, and mining methods and practices necessarily conform to these conditions. The general topography of the coal fields of Iowa is flat to rolling. Many of the coal beds are lenticular and are not continuous. These beds occur under heavy deposits of glacial drift, which usually obscure the outcrop of the beds. However, one bed, the Mystic, is fairly consistent in thickness and extent. The dip of the coal beds ranges from slightly inclining to steeply dipping in some areas. Mines are developed by shafts, drifts, slopes, and stripping, depending upon physical conditions. Usually the room-and-pillar system of mining is used in most areas of the State. However, in Appanoose County, where the Mystic bed is mined extensively, the longwall system of mining is followed. (See fig. 4.) Coal beds, lying at shallow depths of 65 feet or less, are being mined successfully by stripping. In 1948, 788,126 tons of coal were produced from strip mines. This represents 47.2 percent of the total production of the State. The output of coal loaded mechanically underground increased from 13.4 percent in 1943 to 22.2 percent in 1948. Mobile loading machines and conveyors comprise the loading equipment.

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[19] Travis, Raymond G., and Turnbull, Louis A., Coal-Mining Methods and Practices and Electric Power Requirements in the Bull Mountain Coal Field, Montana: Bureau of Mines Rept. of Investigations 4684, 1950, 27 pp.

[19a] See footnote 10.

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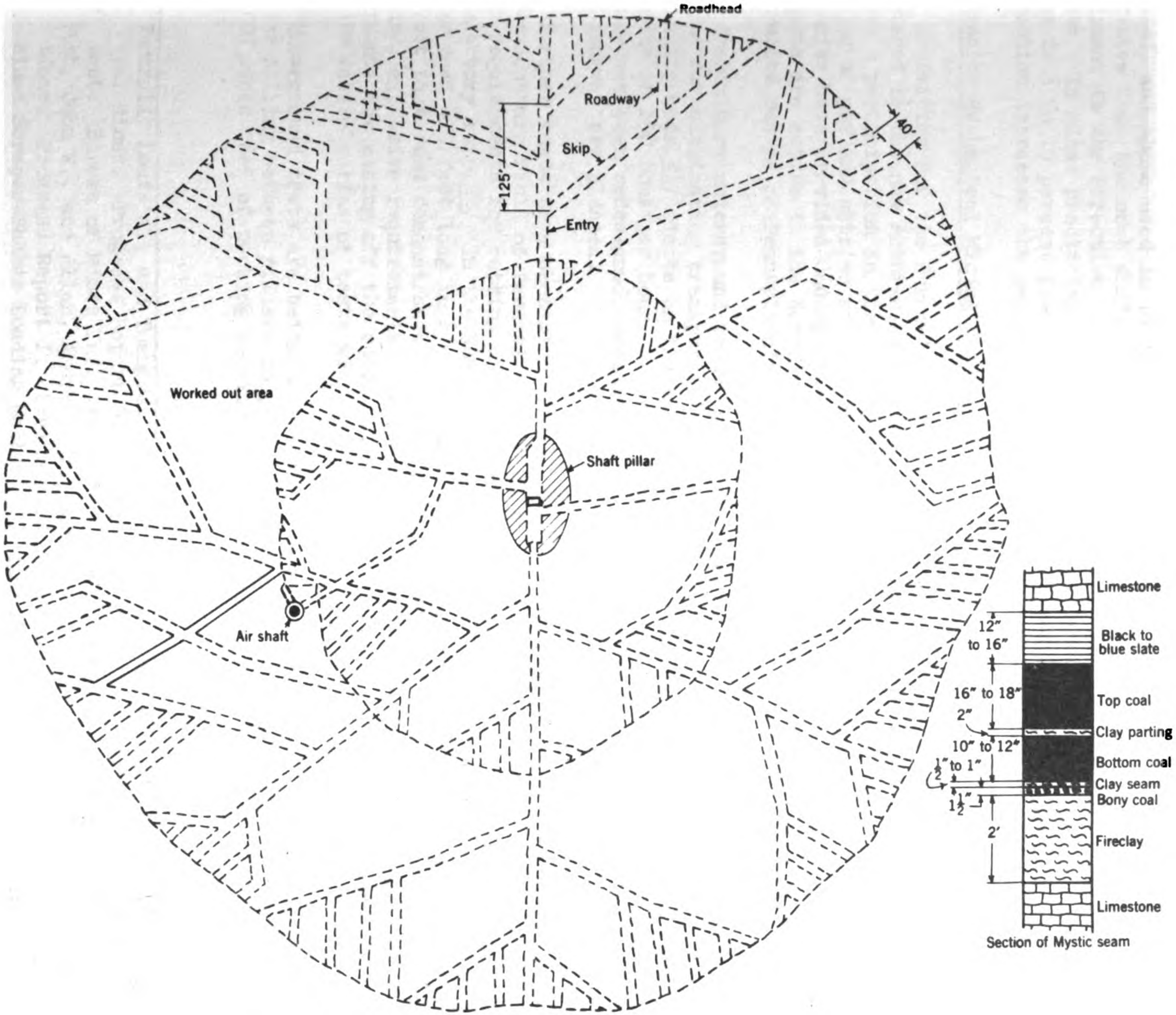

Figure 4. - Map of an Iowa longwall mine, Centerville district.

Mechanical Mining Studies

The methods and practices followed in extracting pillars with mechanized equipment at 55 coal mines in all of the important coal fields of the United States were studied, and those used in 10 of the mines were described.[20] This study clearly indicates that the most difficult problem in extracting pillars with mechanized equipment is the effective control of the immediate roof and the entire overlying strata. In mines practicing pillar extraction, production per mobile loading machine is 6 to 10 percent less than in mines not practicing such extraction. Such extraction increases the percentage of recovery of coal in a given area.

Anthracite Mechanical Mining Research

Activities for the Bureau's anthracite mechanical mining studies are now headquartered in the new Anthracite Research Laboratory at Schuylkill Haven, Pa. The Bureau's participation in these studies has consisted of design, manufacture, and testing of new and untried mining machinery and engineering services. Cooperating companies have provided underground test sections, labor, power, and supplies. Projects are active in the Northern, Western Middle, and Southern fields of the anthracite region of Pennsylvania.

Preliminary underground tests have been completed on a scraper-shaker loading machine for eliminating transportation delays in developing thin, steeply pitching anthracite beds.[21] Tests on the machine in a rock gangway demonstrated a maximum capacity of 150 tons per hour. Average rated capacity is 70 tons per hour. The machine has been redesigned, reconstructed, and electrified, and further tests in a coal gangway are planned.

Further tests have been made on air-powered German lightweight shearing machines. In the Southern field of Pennsylvania anthracite tests have been completed that show power requirements for cutting of less than 2 horsepower when making shear cuts at a satisfactory rate.[22] In the Northern field tests have been completed for driving a pillar road 420 feet long in a coal bed 28 feet thick, without shattering the pillar. The best test runs demonstrated that, by cutting both horizontals and shears with the machine, explosive requirements were reduced by some 900 percent, as compared to requirements for mining off the solid. The average reduction in explosive requirements for the entire series of tests was 237 percent.

Underground tests are being made with a pneumatic packing machine of German origin for filling between pillars to permit recovery of the pillar coal. To date, over 100,000 cubic feet of packing material has been placed.

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[20] Turnbull, Louis A., and Toenges, Albert L., Mechanical Mining in Some Bituminous-Coal Mines. Progress Report 5. Extraction of Pillars with Mechanized Equipment: Bureau of Mines Inf. Circ. 7527, 1949 59 pp.

[21] Buch, John W., and Allan, Andrew, Jr., Anthracite Mechanical Mining Investigations. Progress Report 1. Preliminary Underground Tests of the Bureau of Mines Scraper-Shaker Loading Machine for Driving Gangways: Bureau of Mines Rept. of Investigations 4500, 1949, 9 pp.

[22] Buch, John W., and Allan, Andrew, Jr., Anthracite Mechanical Mining Investigations. Progress Report 2. Preliminary Testing of Eickhoff Shearing Machine, Model DEK: Bureau of Mines Rept. of Investigations 4501, 1949, 14 pp.

Measurements of loads borne by roadway supports were made as a basis for the design of a telescopic working-face shield for more efficient and safer roof support. Underground mine timbers were removed, and their physical properties were determined. Plans are under way and instruments are being built to measure electrically the actual underground roof loads in gangways.

Experimental work is being conducted in the design of precast, reinforced concrete timber sets for use in permanent-type roof supports in gangways. These sets are less affected by underground elements and are made with a slate aggregate, thereby utilizing an anthracite byproduct which formerly was discarded.

Compressed-Air Receivers

The important advantages of large-capacity compressed-air receivers over standard receivers in underground mining operations were summarized.23/ These advantages include: (1) Elimination or reduction of underground work stoppages due to compressor breakdowns; (2) increased efficiency due to constant, full air pressure at the machines using the air; (3) possible elimination of some existing compressor capacity with a better load factor for that remaining; and (4) cooling of the compressed air by storage for a period of time in the receiver.

Studies on the Use of Diesel Engines Underground

In connection with the evaluation of liquid fuels and their behavior in Diesel engines, a study of the composition of Diesel exhaust gas was carried out in detail. Such knowledge is essential when Diesel engines are used underground and is helpful in the solution of many combustion and operation problems.24/

The exhaust gases from a Diesel engine consist chiefly of carbon dioxide, water vapor, nitrogen, and excess oxygen. The constituents of Diesel exhaust gas that are harmful to health at certain concentrations are carbon dioxide, carbon monoxide, oxides of nitrogen, and oxides of sulfur (if the fuel contains sulfur); aldehydes are objectionable because of their odor and smoke and of their contaminating effect in the atmosphere and in the engine. When Diesel engines are operated underground or in a confined space, ventilation should be provided to maintain the concentration of any constituent at the allowable values.

The composition of Diesel exhaust gas depends chiefly on the fuel-to-air ratio. However, the relation between fuel:air ratio and the constituents present in low concentrations (carbon monoxide, aldehydes, and oxides of nitrogen) is affected to some extent by type of engine, composition of the ambient atmosphere in which the engine operates, barometric pressure, and cetane number of the fuel. This relation is affected only slightly over wide ranges of pressures in the intake and exhaust manifolds.

Carefully controlled operation of a Diesel engine showed that at fuel:air ratios greater than the stoichiometrical or chemically correct value (that is, operation in a deficiency of air) the concentration of products of incomplete combustion (carbon monoxide, hydrogen, methane, and unburned carbon) increased rapidly with an increase in fuel:air ratio. Operation of a Diesel engine under such conditions is not normal and is always accompanied by the production of excessive quantities of smoke.

23/ Allan, Andrew, Jr., Summary of Published Information on Large-Capacity Compressed-Air Receivers for Underground Mines: Bureau of Mines Inf. Circ. 7541, 1950, 7 pp.

24/ Elliott, M. A., Combustion of Diesel Fuel: Quarterly Trans. Soc. Auto. Eng., vol. 3, July 1949, pp. 490-515.

Attempts to remove aldehydes from Diesel exhaust gas by scrubbing with water were not completely successful. However, aqueous sodium sulfite solution, inhibited by the addition of hydroquinone to prevent oxidation of the sulfite, removed substantially all of the aldehydes and effected a significant reduction in odor and irritation for extended periods of time.

The Bureau of Mines work on the use of Diesel engines for underground haulage was reviewed before the Fifth International Conference of Directors of Mine Safety Research.25/

Coal-Mining Practices in Germany and Japan

Coal mines in the Ruhr district of Germany were opened decades ago, but progress in developing modern haulage systems has been handicapped by the cost of the changes needed to accommodate more modern equipment. Some changes that have been made are the use of larger locomotives and the adoption of skip hoisting, but notable advances have been made only at newer plants.26/

The Bureau of Mines furnished safety engineers to the UK/US Coal Control Group supervising postwar production from the Ruhr district of western Germany. In connection with their investigation of safety conditions, some of the operating conditions and practices also were studied. Coal beds in the active field of operations are far from exhausted, and preparation plants are generally efficient.27/ Virtually all coal in Germany is mined by the longwall system, but only a small amount of the total production is mined mechanically. Not more than 6 percent is blasted. Ventilation systems are efficient in removing methane, but temperatures were high in many mines.

Safety practices, mining conditions, and methods of mining were studied for the purpose of making recommendations for the revision of the Coal-Mine Safety Standards for Japanese mines. Geological conditions, mining and safety practices, and accident rates of the principal coal fields in Japan are described separately.28/

Control of Coal-Mine Fires

The control and extinguishment of mine fires is under investigation by the Bureau, both in actual occurrences and in test procedure at the Experimental Mine. Through trials and tests, attempts are made to find effective methods and equipment, particularly for use against coal-mine fires. Sealing, flooding, and use of carbon dioxide have been the methods applied in fighting recent mine fires; in some instances, the three methods were used in combination. Carbon dioxide was effective in an instance where fire seals would not hold the pressure of water to flood the

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25/ Elliott, Martin A., Review of Bureau of Mines Work on Use of Diesel Engines Underground: Bureau of Mines Bull. 489, 1950, pp. 140-167.

26/ Stahl, R. W., Sanford, H. E., and Benson, J. B., Underground Transportation in the Ruhr Coal-Mining District of Germany: Bureau of Mines Inf. Circ. 7547, 1950, 9 pp.

27/ Benson, J. B., Sanford, H. E., and Stahl, R. W., Conditions and Practices at Coal Mines in the Ruhr District of Western Germany: Bureau of Mines Inf. Circ. 7549, 1950, 48 pp.

28/ Warncke, Russell G., Observations of Safety Practices and Conditions in Japanese Coal Mines: Bureau of Mines Inf. Circ. 7542, 1949, 38 pp.

enclosed area but could hold the gas.[29] In another instance, carbon dioxide introduced into a sealed area cooled the area sufficiently to permit prompt reopening without immediate rekindling of the fire, although the gas leaked through the seals into abandoned areas and did not extinguish the fire. The hot material was loaded out with a loading machine, rock dust being applied to shield men and equipment from the extremely hot rock and slag.[30]

Decreasing Explosive Hazards Underground

Since its introduction into coal mines, the use of Airdox has grown in popularity, especially in the Indiana and Illinois coal fields. Many of the hazards connected with the use of explosives are eliminated, but when Airdox lines are electrically grounded as specified by the Federal Mine Safety Code, there is danger of arcing through contact with mine tracks or other mine equipment. To eliminate such arcing, the use of reinforced rubber-jacketed tubing and insulating air-line couplings has been introduced in coal mines where Airdox is used for breaking coal.[31] Premature blasts of charges of explosives have caused many deaths in anthracite mines, and the causes of many of the blasts were not determined satisfactorily. Investigation often has found fairly high potential differences between rails, pipes, conveyor lines, or sheet-iron chutes in working places where these accidents occurred, and it is considered probable that these potential differences have caused premature firing of the electric detonators. A survey of a large number of such incidents indicated that this hazard could be reduced by proper bonding of metallic installations.[32]

Toxic Mine Atmospheres

Conditioning air is one of the most essential factors to be considered in underground workings, not only from the standpoint of health and safety, but also with regard to efficiency and productivity. As an instance, mechanical refrigeration cooled off new lower levels in copper mines in a month where 3 years would have been required for cooling by ventilation with the normal mine air. To survey the theoretical and practical methods of conditioning air in mines and the results obtained by some of the systems now in use, a review of published information has been made.[33]

Although dust control is practiced in many bituminous-coal and lignite mines, the extent and effectiveness of control over the entire industry is not generally known. Factual data on dust-allaying practices at working faces in a large number of the bituminous coal and lignite mines in the United States were compiled from reports on Federal inspections of these mines.[34] Although dust produced at faces was allayed effectively in about one-third of the mines requiring dust allaying, no effort was made to control such dust in more than half of these mines. The allaying of dust at the source in bituminous-coal mines will reduce the dust-explosion hazard and protect men from respiratory infection from inhaling coal dust.

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[29] Westfield, James, Brumbaugh, H. C., and Whittaker, R. W., Extinguishing Fire with Carbon Dioxide in the Valier Mine, Valier Coal Co., Valier, Franklin County, Ill.: Bureau of Mines Inf. Circ. 7563, 1950, 10 pp.

[30] O'Connor, J. A., Malesky, J. S., and Higgins, T. C., Fighting a Fire in No. 59 Mine, Peabody Coal Co., Springfield, Sangamon County, Ill.: Bureau of Mines Inf. Circ. 7564, 1950, 19 pp.

[31] Gallagher, F. J., Two Devices to Prevent Electric Arcs with Airdox Operations in Coal Mines: Bureau of Mines Inf. Circ. 7515, 1949, 5 pp.

[32] Weber, Charles F., A study of Stray Currents in Pennsylvania Anthracite Mines: Bureau of Mines Rept. of Investigations 4637, 1950, 8 pp.

[33] Forbes, J. J., Davenport, Sara J., and Morgis, Genevieve, Review of Literature on Conditioning Air for Advancement of Health and Safety in Mines. Part III. Methods of Controlling the Chemical and Physical Qualities of Underground Air: Bureau of Mines Inf. Circ. 7528, 1949, 57 pp.

[34] Forbes, J. J., Franklin, R. K., and Reese, S. T., Review of Dust-Allaying Practices at Working Faces in Some Bituminous-Coal and Lignite Mines: Bureau of Mines Inf. Circ. 7566, 1950, 29 pp.

In connection with the work carried out under the provisions of the Federal Coal-Mine Inspection Act, more than 16,000 samples of mine air were analyzed to determine the adequacy of ventilation in coal mines, to detect and aid in the elimination of hazards from flammable and toxic gases, and to aid in the control and extinguishment of mine fires.

Surveys were conducted in coal mines to determine the concentrations of air-borne dust produced by various mining operations, thus to provide information for the formulation of recommendations for control measures leading to the reduction of exposure of coal-mine workers to harmful dusts.

The production of dust by roof-drilling for roof bolting in coal mines was studied. As the roof strata usually contain a much higher percentage of free silica than the coal measures, the problem of control of the dust produced by roof-drilling is a serious one for those engaged in such drilling operations. A test program was initiated to study the efficacy of various devices that have been developed commercially to control or trap the dust produced by such operations.

More than 1,000 examinations were conducted to determine the concentration and particle size of air-borne dusts and the composition of such dusts and dust-source materials to evaluate their hygienic significance in the working environment of coal and other types of mines. These examinations were conducted by microscopic, petrographic, X-ray diffraction, and spectrographic methods. Examinations were also made of various materials to determine their suitability for use in rock-dusting coal mines to reduce the possibilities of coal-dust explosions.

Studies relating to the determination of air-borne dust were described in two publications issued during the year.[35][36] Studies were also conducted of dust dissemination by the use of different-type treads on tires of coal-mine shuttle cars, and the attendant compacting of the mine roadways was observed.[37]

Approval testing was conducted on respiratory protective devices, including gas masks and dust respirators applicable to use for personal protection in the mining and mineral industries. Eight new approvals and 25 extensions of approval of such devices were granted, representing modifications and improvements in design. In many instances such modifications were developed in a form suitable for Bureau of Mines approval only after extended tests and consultations between Bureau personnel and the manufacturers of the respiratory protective devices. To aid industry in the selection of respiratory protective devices and to present up-to-date information on the subject, a listing was prepared of all such devices approved by the Bureau of Mines.[38] Because of increasing interest in the control of and protection against dust produced in coal-mining operations, the use of dust respirators in this application was discussed in a publication which includes instructions on the selection, care, and maintenance of these devices.[39]

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[35] Foster, Wilder D., Petrographic Determination of Quartz in the Presence of Clay-like Minerals: Bureau of Mines Rept. of Investigations 4573, 1949 13 pp.

[36] Brown, Carlton E., and Schrenk, H. H., Relation Between and Precision of Dust Counts (Light- and Dark-Field) from Simultaneous Impinger, Midget-Impinger, Electric-Precipitator, and Filter-Paper Samples: Bureau of Mines Rept. of Investigations 4568, 1949, 35 pp.

[37] Nicholas, R. H., Whittaker, J. S., Dornenburg, D. D., Harmon, John P., and Bank, Walter, Shuttle-Car Tire and Roadbed Study: Bureau of Mines Rept. of Investigations 4624, 1949, 22 pp.

[38] Pearce, S. J., and Berger, L. B., List of Respiratory Protective Devices Approved by the Bureau of Mines: Bureau of Mines Inf. Circ. 7570, 1950, 16 pp.

Mine ventilation studies conducted by the Bureau were coordinated with new developments in this field as applied by the coal-mining industry. A historical résumé was prepared, outlining studies conducted by the Bureau of Mines on mine and tunnel ventilation during the period 1910 to 1949.[40]

The ventilation-survey practice of a large anthracite producing company in South Wales in Great Britain was reviewed[41] to provide background material for American colliery managers who are planning to make ventilation surveys as a step toward improving environmental conditions in their mines. This British company is staffed to make such surveys as a routine operation. Rates of air flow in underground air current are measured on a circuit basis with rotating vane anemometers, and pressure changes are measured on inclined U-tube manometers. Results are plotted on the mine map so that zones of excessive loss are revealed instantly.

A two-color psychrometric chart, especially adapted to solving mine air-conditioning problems at all barometric pressures, was published.[42]

Roof Control

Early in 1947 the Bureau of Mines became interested in adapting suspension roof support to coal mining as a possible means of reducing the high rate of accidents from falls of roof. For a time the program made little progress because of the shortage of steel products after the end of World War II, but with the easing of the shortage and as of May 1, 1949, experimental work had either been started or materials and equipment in anticipation of trial installations had been ordered at 114 mines. It is estimated that 30 linear miles of roof in underground passageways have been secured by this method in recent months, and no lost-time roof-fall accident has been reported in these areas up to that date.

The Bureau of Mines, in cooperation with several mining companies and State agencies, is carrying on a program of research and investigation not only on problems involving suspension roof supports, but also on the broader subject of roof control in general.[43]

Anthracite Flood Prevention

The Bureau of Mines continued its work in obtaining and evaluating information on the underground mine-water problem in the anthracite region of Pennsylvania and in preparing plans that would aid in solving this problem. In the course of this work the application, design, and performance of deep-well and shaft pumps were studied for possible use in a project to pump underground waters.[44]

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[39] Pearce, S. J., The Use of Dust Respirators in Coal Mines: Bureau of Mines Inf. Circ. 7561, 1950, 6 pp.

[40] McElroy, G. E., Historical Résumé of Mine and Tunnel Ventilation Studies, Bureau of Mines, 1910-49: Bureau of Mines Inf. Circ. 7556, 1950, 16 pp.

[41] Smith, Cloyd M., Routine Ventilation Surveying in South Wales Anthracite Mines: Bureau of Mines Inf. Circ. 7530, 1949, 11 pp.

[42] McElroy, G. E., A Mine Air-Conditioning Chart: Bureau of Mines Rept. of Investigations 4165, 1947, 23 pp. (Chart reproduced on larger scale in 1949.)

[43] Thomas, Edward, Barry, A. J., and Metcalf, Arthur, Suspension Roof Support. Progress Report 1: Bureau of Mines Inf. Circ. 7533, 1949, 13 pp.

An engineering study of the inundated anthracite reserves of the Eastern Middle field of Pennsylvania and of the various methods that can be used to unwater these reserves was made.[45] One of the major methods of handling mine water at the anthracite mines is by pumping. Data on the types of pumps and their performance in the Pennsylvania anthracite region were presented, with the relationship between precipitation, anthracite production, and the amount of water pumped to the surface.[46]

Other investigations that are being conducted on the problem of underground water in the anthracite region of Pennsylvania include studies of centrifugal-pump installations in the anthracite mines of Pennsylvania, the buried valley of the Susquehanna Valley in the region, a plan for dewatering the mines by driving a drainage tunnel, the strength of barrier pillars underground, and estimation of the tonnage of anthracite that would be affected by any drainage method.

PREPARATION OF COAL

Coal Washing

Cyclone Thickener Performance Tests

Under a cooperative agreement with the Truax-Traer Coal Co., Chicago, Ill., the Bureau of Mines installed and operated a 14-inch-diameter cyclone thickener at the preparation plant of the Shamrock mine, Kayford, W. Va.[47] The unit was placed in the fine-coal washery circuit and used as a water-clarification device. Test data indicate the recovery of substantially all coal particles 100-mesh and larger in size. Less efficient recovery is experienced as the particle size decreases, with but a one-third recovery of those solids finer than 325-mesh.

This one cyclone recovered 5 to 6 tons per hour of marketable coal, which was previously lost. The demonstration of the usefulness of this device in the preparation plant influenced the cooperating company to install three additional cyclones to handle the entire water volume of the fine-coal washery circuit.

Coal-Preparation Bibliography

To meet the many requests for information on the preparation of coal, a bibliography of Bureau of Mines publications on the subject was compiled.[48]

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[44] Lesser, William H., Deep-well Pumps and Shaft Pumps in Anthracite Mines of Pennsylvania: Bureau of Mines Rept. of Investigations 4656, 1950, 52 pp.

[45] Ash, S. H., Kynor, H. D., Fatzinger, R. W., Davies, B. S., and Gilbert, J. C., Inundated Anthracite Reserves: Eastern Middle Field of Pennsylvania: Bureau of Mines Bull. 491, 1950, 28 pp.

[46] Ash, S. H., Eaton, W. L., Gilbert, Joseph C., James, H. M., Jenkins, Hayden E., Kennedy, D. O., Kynor, H. D., Link, Howard B., and Romischer, W. M., Data on Pumping at the Anthracite Mines of Pennsylvania: Bureau of Mines Rept. of Investigations 4700, 1950, 264 pp.

[47] Fraser, T., Sutherland, R. L., and Giese, F. F., Performance Tests of an Experimental Installation of Cyclone Thickeners at the Shamrock Mine: Am. Inst. Min. and Met. Eng. Tech. Pub. 2615F, Min. Trans., vol. 184, 1949, pp. 436-446; Min. Eng., vol. 1, December 1949, pp. 439-446.

[48] Crentz, William L., Publications of the Bureau of Mines on Coal Washing: Bureau of Mines Inf. Circ. 7531, 1949, 7 pp.

Preparation Characteristics of Illinois Coals

As part of the Bureau's survey of preparation characteristics of American coals, an examination was made of the coals of Illinois.[49] Float-and-sink tests and screen analyses of representative samples from the important mining districts and beds indicated the comparative responsiveness of these coals to mechanical preparation.

The recoverable clean coal readily obtained by conventional coal-washing practices operating at a separation gravity of about 1.60 contained 6.0 to 13.0 percent ash. Test separations at 1.30 specific gravity showed many coals had fractions with as low as 4.0 percent ash, but the quantity of this low-ash coal is generally less than half the original raw coal.

Much of the Illinois coal is high in sulfur, some exceeding 5 percent, but there are areas which are singularly low in sulfur content in both the raw and washed coal. The low sulfur and ash contents of the No. 6 coal bed in the Franklin-Williamson mining district is well-known and frequently has been examined as a possible source of metallurgical fuel. Tipple samples of nut and egg sizes from the No. 5 bed in Saline County and the No. 6 bed in Jefferson County showed substantial yields of low-sulfur float coal, and face samples taken in these mines corroborate the feasibility of recovering float coal containing a sulfur content within the present tolerances for metallurgical use. Face samples from the No. 2 bed in northern Illinois showed some coal of this category in the finer sizes.

With regard to the size consist of screenings, a factor of major importance in appraising an industrial fuel, a substantial increase in fines occurred during the period 1914 to 1935, with only a very small change since.

The finer sizes of Illinois coals are more responsive to ash reduction by specific-gravity separation. On the other hand, even fine crushing of high-sulfur raw coal does not materially effect the release of sulfur so as to permit the recovery of a low-sulfur coal by mechanical cleaning.

Preparation Characteristics of Cumberland Plateau, Tenn. Coal

A washability study was completed on one of the coal beds in the Cumberland Plateau of Tennessee.[50] Data were compiled from laboratory tests on the coal, and a discussion of the results to be expected from the treatment of this coal by conventional coal-preparation methods. It was found that if this coal was washed by conventional methods, such as jigging and tabling, a product could be obtained containing approximately 12.3 percent ash and 2.07 percent sulfur; however, if by the use of heavy-medium methods separations between washed coal and refuse could be made at specific gravities as low as 1.40 on the sizes above three-sixteenths inch, a washed coal of about 10.1 percent ash and 1.57 percent sulfur would be obtained.

Upgrading Marginal Coking Coals by Coal-Preparation Methods

In a Nation-wide appraisal of coking-coal reserves and of the feasibility of expanding them by upgrading marginal coals, an investigation was made of the washing characteristics of the Pittsburgh bed coal in Allegheny, Fayette, Washington, and Greene Counties, Pa., and Monongalia, Marion, Marshall, Ohio, Brooks, and Hancock Counties, W. Va.[51] Intensive study of the washability data shows the main washing problem concerns the release of sulfur. This problem is increased by the wide variations of sulfur content not only in the raw coal, but in the float product. No one method of cleaning will result in satisfactory upgrading of the high-sulfur coals to metallurgical standards. Selective mining, low-gravity separation, fine crushing and froth flotation, and combinations of the three must be employed before sulfur can be reduced significantly. Coals containing 1.80 percent sulfur in the raw product present no extraordinary technological problem, while coals containing sulfur in excess of 2.27 percent in the raw state cannot be considered as a metallurgical fuel. When the sulfur content of the raw coal is in the 1.81 to 2.27 percent range, the Pittsburgh-bed coal in the area studied may be upgraded to metallurgical standards by the development and application of intensive preparatory treatment.

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[49] Crentz, William L., Preparation Characteristics of Illinois Coals: Bureau of Mines Tech. Paper 724, 1949, 112 pp.

[50] Gandrud, B. W., and Riley, H. L., Washability Study of Coal from a Strip Pit Bed on the Cumberland Plateau, Near Chalybeate, Van Buren County, Tenn.: Bureau of Mines Rept. of Investigations 4596, 1949, 10 pp.

Coal Preparation for Synthetic Fuels

A study was made of the effect of ash on oil losses during the elimination of nonliquefiable portions of the coal in the hydrogenation process.[52] The oil loss increases with ash content and results in increased consumption of coal, hydrogen, and power. To determine the economic limits for cleaning coal for hydrogenation due to this one factor, a comparison was made of the claculated economic advantages resulting from the use of a 2.3 percent ash coal from Rock Springs, Wyo., and a 6.0 percent ash coal from the same mining field in a 30,000-barrel-per-day hydrogenation plant.

Table 2 shows the maximum allowable cleaning costs in cents per ton of coal as received for each percent of ash reduction, using coals containing 10, 20, and 40 percent ash plus moisture and a washery recovery of 50 and 70 percent of the raw feed as clean coal. A large proportion of the remaining 50 and 30 percent, respectively, may be recovered as middlings and utilized as powerhouse coal. The calculations reflect the decreased fuel value of this middling product.

TABLE 2. - Maximum allowable washing costs for different coals for each unit of ash reduction, cents per ton of raw coal, as-received basis

Yield of washed coal .... percent	50			70		
Ash plus moisture in raw coal as received ..... percent	10	20	40	10	20	40
Maximum allowable cost of washing for each unit of ash reduction, cents per ton:						
Based on moisture- and ash-free coal valued at $2.00 per ton (7 cents per million B.t.u.)	3.8	3.4	2.5	5.3	4.7	3.5
Based on moisture- and ash-free coal valued at $4.00 per ton (14 cents per million B.t.u.)	4.5	4.0	3.0	6.3	5.6	4.2
Based on moisture- and ash-free coal valued at $6.00 per ton (21 cents per million B.t.u.)	5.1	4.6	3.4	7.5	6.6	5.0

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[51] Fraser, Thomas, Crentz, W. L., and Bailey, A. L., High-Sulfur Pittsburgh Coal: Upgrading in Southwestern Pennsylvania and Northern West Virginia: Bureau of Mines Bull. 483, 1950, 70 pp.

[52] Crentz, W. L., Doherty, J. D., and Donath, E. E., Coal Preparation for Synthetic Liquid Fuels: Am. Inst. Min. and Met. Eng. Tech. Pub. 2832F, Trans. Am. Inst. Min. and Met. Eng., vol. 187, 1950, pp. 507-510; Min. Eng., vol. 187, April 1950, pp. 507-510; discussion, Trans. Am. Inst. Min. and Met. Eng., vol. 187, 1950, p. 1158; Min. Eng., vol. 187, Sec. 1, November 1950, p. 1158.

Using a cost figure of 15 cents per ton of coal feed to the washery as the operating cost of the washing plant, it is possible to calculate the minimum reduction in ash content for different coals to justify washing the coal. These data appear in table 3.

TABLE 3. - Minimum reduction in ash content to justify a washing cost of 15 cents per ton of raw coal, percent

Yield of washed coal .... percent	50			70		
Ash plus moisture in raw coal as received .... percent	10	20	40	10	20	40
Reduction in ash content necessary to justify a washery cost of 15 cents per ton of feed coal:						
Based on moisture- and ash-free coal valued at $2.00 per ton (7 cents per million B.t.u.)	4.0	4.4	6.0	2.8	3.2	4.3
Based on moisture- and ash-free coal valued at $4.00 per ton (14 cents per million B.t.u.)	3.4	3.8	5.0	2.4	2.7	3.6
Based on moisture- and ash-free coal valued at $6.00 per ton (21 cents per million B.t.u.)	2.9	3.2	4.3	2.0	2.3	3.0

Briquetting of Coal

The Bureau continued cooperative work with the Natural Resources Research Institute, University of Wyoming, on problems incident to briquetting of subbituminous coal. Fuels for briquetting were prepared and dried at the Bureau laboratories and then briquetted in the pilot plant at the University. A publication describing this investigation was issued by the University.[53]

During the year, an integrated pilot plant for drying and briquetting coal was designed by the Bureau and erected at the University of Wyoming by the Natural Resources Research Institute. This pilot unit for processing coal at the rate of about 3 tons per hour is illustrated in figure 5. The fluidized drying process developed by the Bureau will dry and preheat the subbituminous coal fed to the briquetting press.

Drying Low-Rank Coals

Rocky Mountain Coals

Investigations on the drying of low-rank coals in the entrained and fluidized state were continued[54] in the small pilot plant illustrated in figure 6. Various grades and sizes of coal up to three-fourths inch were studied in this unit. It was found that, when drying any surface-dry low-rank coal having natural bed moisture ranging from 15 to 65 percent, the major operating factor was to strike a heat balance to accomplish the required drying. It was proved that theoretical and practical operation of the drying process coincided and the performance of the dryer could be estimated in advance. This important confirmation allows correct appraisal of the drying of surface-dry coals without the formality of testing. It was further

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[53] Boley, Charles C., and Rice, Neal, Briquetting of Dried Low-Rank Western Coals: Univ. Wyoming Bull. 3, 1949, 76 pp.

[54] Parry, V. F., and Wagner, E. O., Upgrading Missouri Basin Coals by Flash Drying: Colorado School of Mines Quarterly, vol. 45, No. 2B, April 1950, pp. 57-66.

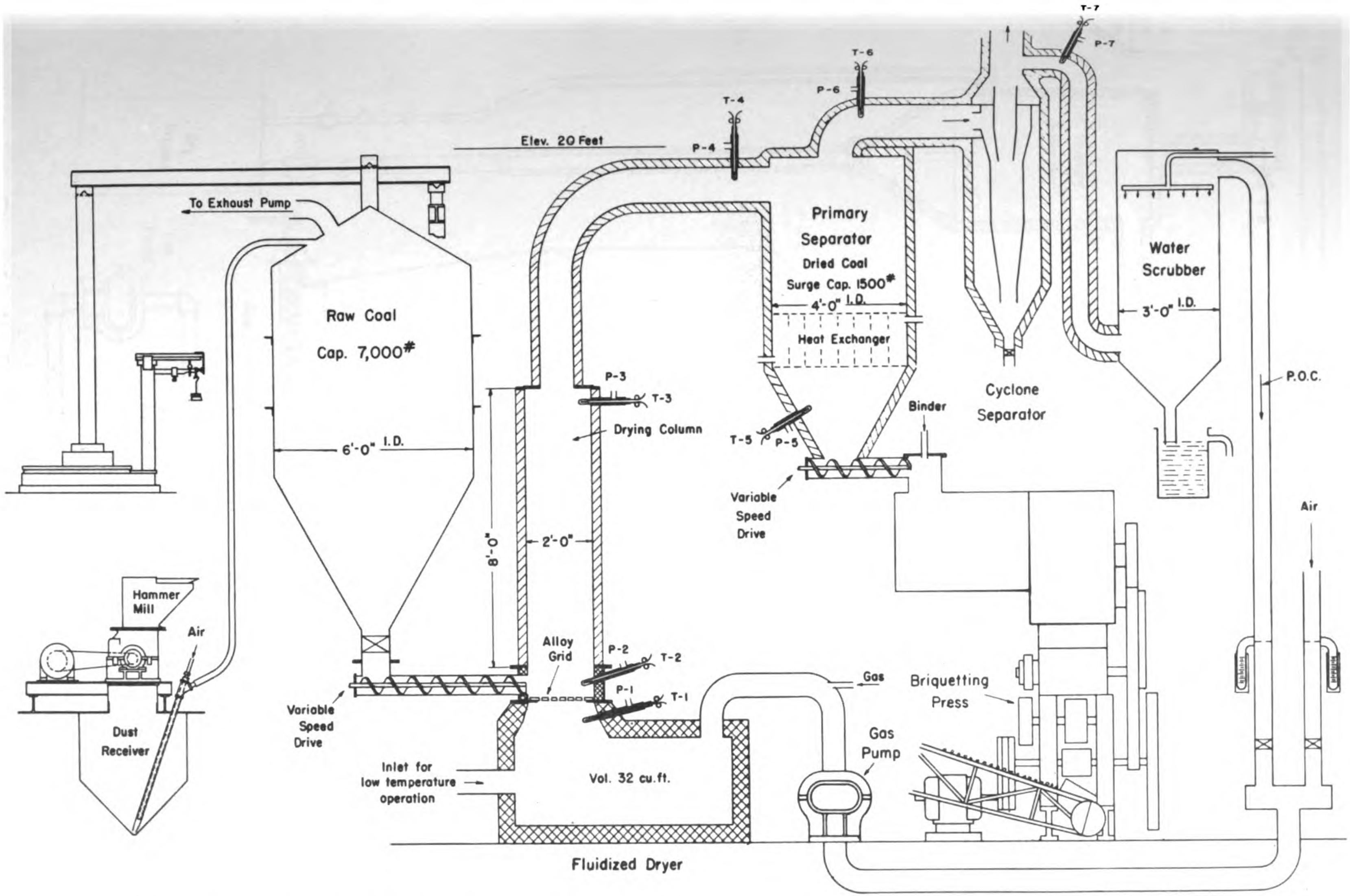

Figure 5. - Integrated pilot plant for fluidized drying and briquetting of coal; capacity 3½ tons raw coal/hr.

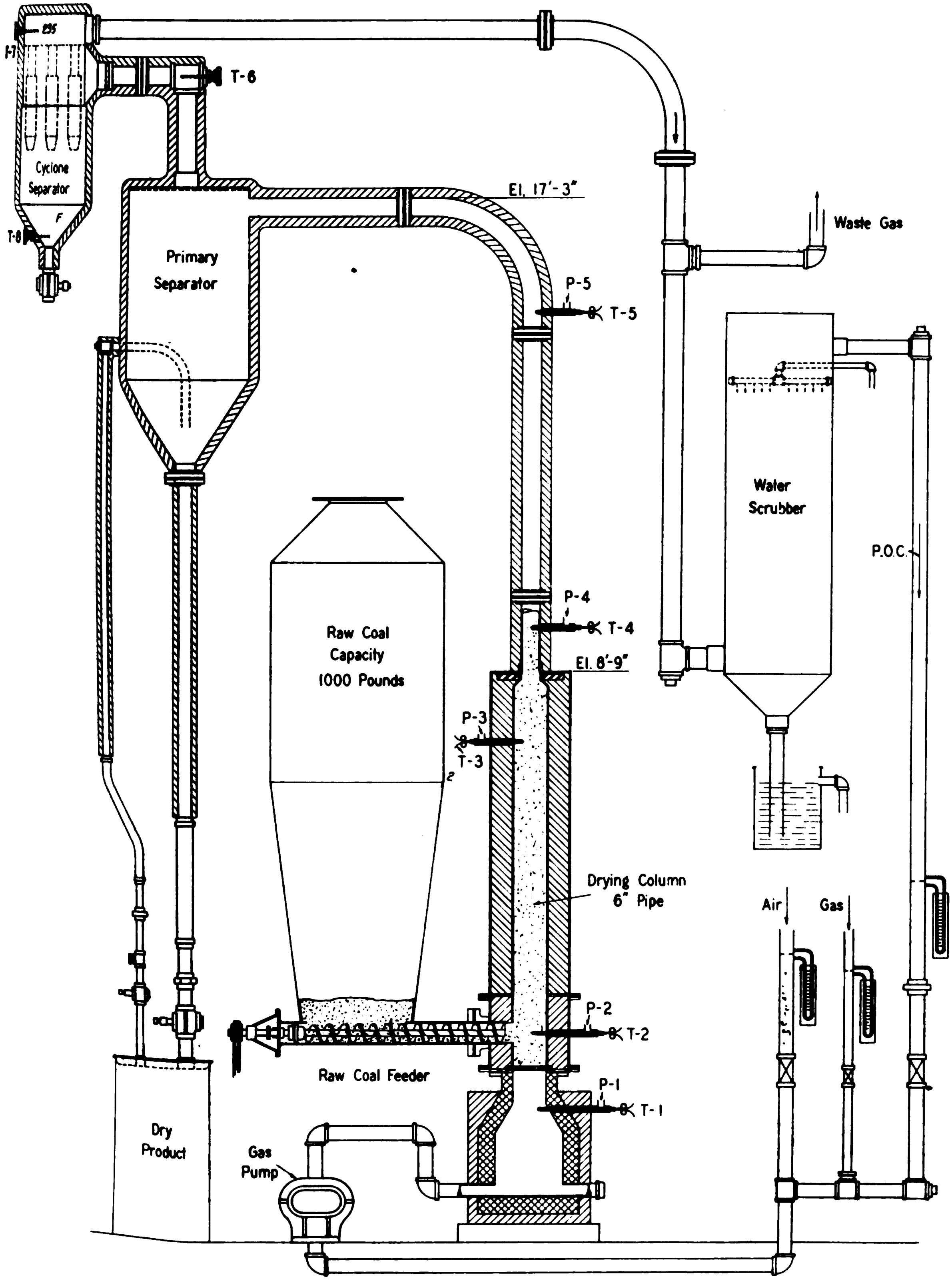

Figure 6. - No. 5 pilot plant for drying coal, Golden, Colo.

observed that the most practical size of raw coal for drying in the entrained state is 1/4 by 0 inch, but larger sizes can be handled as the natural bed moisture increases.

Lignite

American lignites are differentiated from solid fuels of higher rank by their higher natural bed-moisture content of 35 to 40 percent. To gain efficiency in utilization and economy in transportation, it is highly desirable to reduce this moisture content to 15 percent or less. During the year, the Lignite Pilot Plant at Grand Forks, N. Dak., dried 250 tons of lignite for the quadruple purpose of improving the drying operation, continuing storage observations, making combustion tests, and gasification. Table 4 gives the pertinent data and indicates the improvement obtained through drying.

TABLE 4. - Typical analysis of Dakota Star lignite

	Before drying	After drying
Moisture .... percent	37.2	17.4
Improvement ratio	-	1.32
Heat value.......................... B.t.u./lb.	6,930	9,110

Operations were carried out in large autoclaves, using a modified Fleissner process with steam at a nominal pressure of 400 pounds per square inch. This was reduced from 1 hour to 30 minutes, thus doubling the capacity of the plant and cutting fixed charges in half. Approximately the same amount of water (65.0 and 67.5 percent of the total) was removed during the shorter cycle as during the longer one.

A summary of experiments on the drying of lignite in hot oil was published.[55]

Storage of Coal

Low-Rank Rocky Mountain Coals

Field investigations on storage of low-rank coals were continued. Coal operators in the Denver, Colo., area are considering the advantages to be gained by storage of coal at industrial plants and mines and are adopting the improved methods of storage recommended by the Bureau. Another model 4,000-ton pile of subbituminous coal was placed at the Denver Federal Center during March 1950. It will be studied to determine conditions of storage during the hot summer months and will be consumed during the winter of 1950.

Two 5,000-ton piles of subbituminous A slack coal, placed in storage in 1948 according to Bureau recommendations, show no indications of heating or deterioration.

Lignite

To date, approximately 350,000 tons of lignite has been placed in storage in uncapped piles at the Garrison Dam in North Dakota. Heating was anticipated at one location because of improper pile-forming practices, and, as expected, a small fire developed at that point. As a result of this fire, the stockpiling contracts were changed to specify the rolling of each 1-foot increment of lignite with a sheep's-foot roller in order to achieve greater fragmentation and compaction. There have been no indications of heating since the introduction of the sheep's-foot rolling technique.

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[55] Burr, Alex. C., Oppelt, Walter H., and Kamps, Theodore W., The Rate of Drying of Lignite in Hot Oil at 315° F.: Proc. North Dakota Acad. Sci., vol. 2, 1949, pp. 32-36.

Figure 7 shows the occurrence of lignite beds 1A and BB in the powerhouse excavation. The northern end of pile 3 appears at the right-hand edge of the picture. Figure 8 shows how the lignite is stripped from the beds by power shovel and loaded into carry-alls for transportation to the pile sites. (Note the size of the lignite lumps.) The lignite is dumped by the carry-all, leveled by bulldozer, rolled with a sheep's-foot roller, with slope of the sides of the pile maintained at about 17°. The lighter mass on the right is similarly piled lignite which has slacked for several days. The slacking aids fragmentation and compaction by the sheep's-foot roller.

Storage of Dried Lignite

The investigation of safe storage conditions was continued. The old storage pile of approximately 25 tons, which has been exposed to the weather for almost 2 years without firing, was repiled. This time, no attention whatever was paid to segregation, and pile fired at several places on the surface.

One hundred and ninety tons of freshly dried lignite was stored in several piles. Observation of these piles led to the conclusion that freshly steam-dried lignite of approximately 15 percent moisture may be safely stored for indefinite periods in piles of 100 tons or less, if the fines are removed and the dried lignite laid down in layers of approximately 18 inches.

UTILIZATION OF COAL FOR COMBUSTION

Fuel-Engineering Service

As in previous years, fuel-engineering service was rendered to Government establishments in the selection and use of fuel and fuel-burning equipment and in the economical use of steam. Total boiler-plant operating-cost comparisons were made for the use of oil, gas, and coal, and the type of fuel-burning equipment recommended for eight projects for the Veterans' Administration. Studies of boiler-plant equipment and operation at the Naval Research Laboratory, Bellevue, D. C., and the Naval Hospital, St. Albans, N. Y., indicated that plans for equipment for these plants could be changed to effect savings, respectively, of $52,000 and $30,000.

At the request of the Atomic Energy Commission, Oak Ridge, Tenn., tests and recommendations were made to eliminate fuel-burning difficulties in the heating plants of a housing development. Final acceptance tests were made of steam-generating equipment at the West Central Heating Plant, Washington, D. C.; changes shown necessary by previous tests improved the performance and reliability of the equipment so that the specification requirements were satisfactorily met. Assistance was given the Federal Works Agency in the design of a high-pressure overfire-air application for a large furnace fired by an underfeed stoker; tests were then made for boiler efficiency and stack-smoke emission, with various nozzle arrangements and amounts of overfire air, which resulted in the controlled elimination of smoke.

Specifications for a new power plant at Shiprock, N. Mex., of the Bureau of Indian Affairs, were studied, and final wordings recommended. A survey was made of

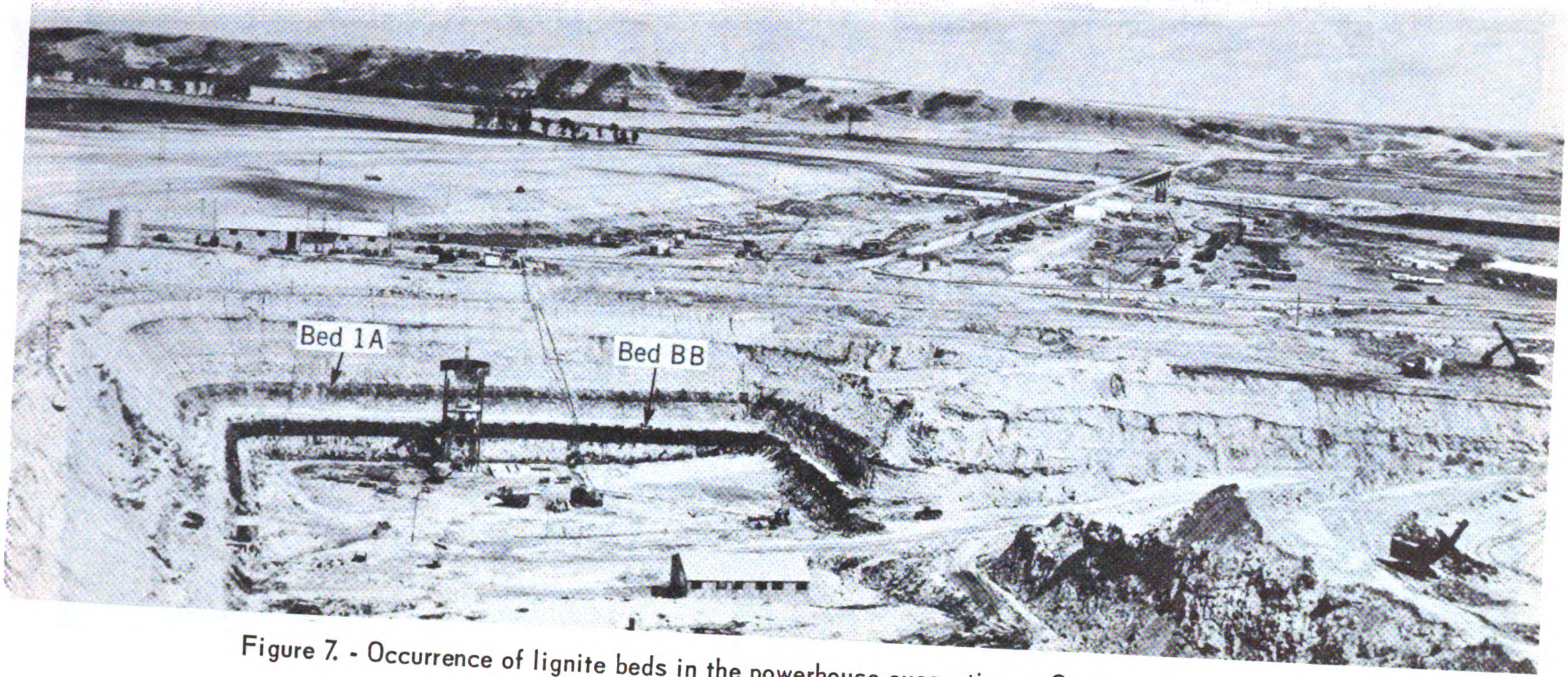

Figure 7. - Occurrence of lignite beds in the powerhouse excavation at Garrison Dam, N. Dak.

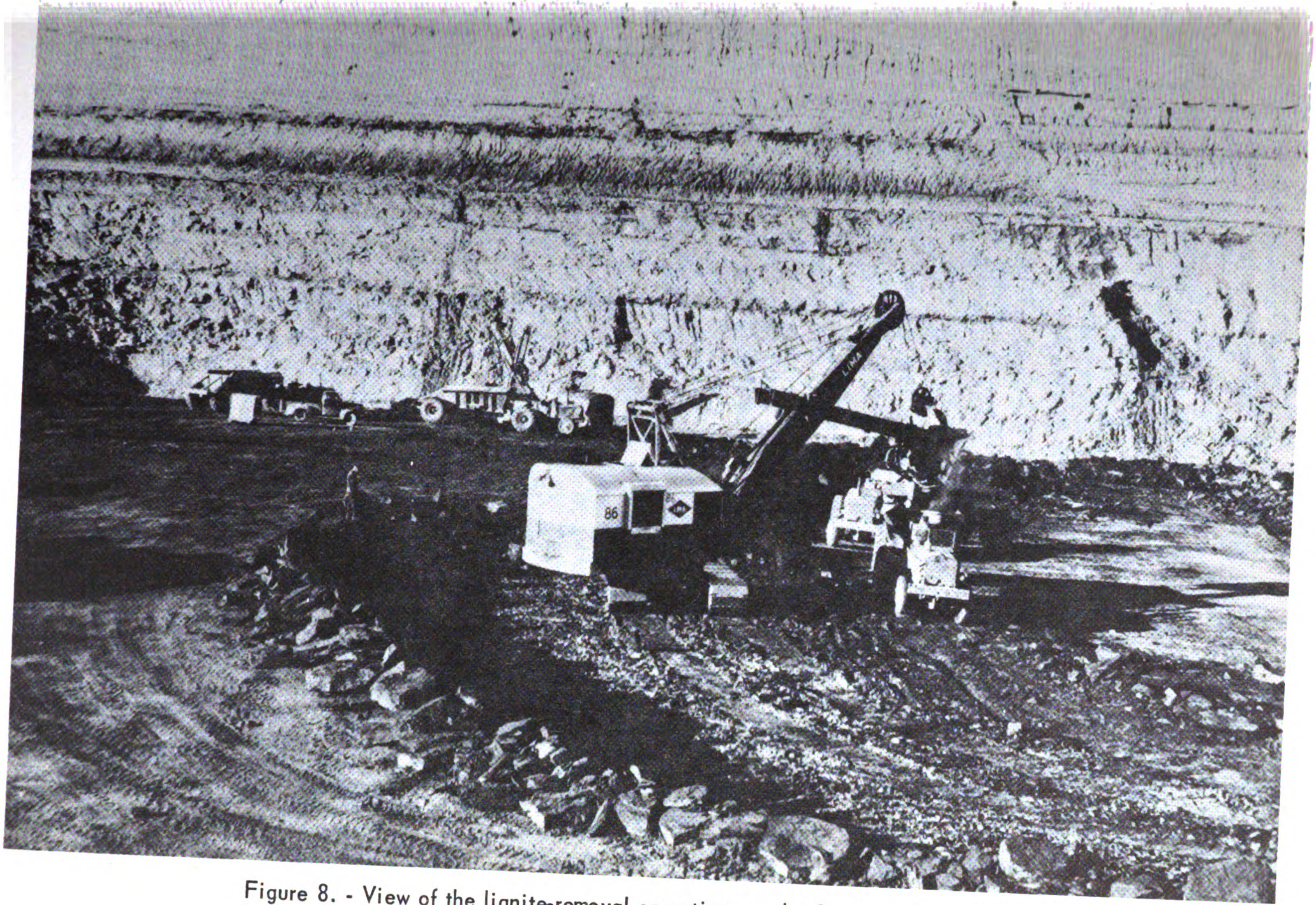

Figure 8. - View of the lignite-removal operations at the Garrison Dam, N. Dak.

a boiler plant of the Veterans' Hospital at Buffalo, N. Y., resulting in recommendations as to operation. Services on 158 special problems were given 32 different Government agencies.

To aid Federal designers of fuel-burning equipment to be installed in cities throughout the United States, a tabulation was made of the design requirements of smoke-abatement ordinances of 22 cities.[56] The tabulation showed that even after many years of fuel-burning practice and experience there is no common standard of design, the variations in the requirements for the same type of equipment under the same conditions of operation being very great.

During research work at the Naval Gun Factory, Washington, D. C., to evaluate the significance of sulfur dioxide and sulfur trioxide in causing corrosion and deposits, it was found that analytical determinations in the gaseous products of combustion for SO_2 and SO_3 as made by the A.S.M.E. code method did not give accurate results. Minute quantities of copper, for example, in the coal being used proved to be one source of the difficulty. Using certain basic procedures of the A.S.M.E. method, new procedures were developed that gave satisfactory results.[57]

A representative of the Bureau of Mines served as chairman of the Bureau of Federal Supply Technical Committee on Solid Fuels which completed a new Federal specification covering the purchase of anthracite. Federal coal-purchasing agents were supplied needed information on mines for use in making awards of contract, the bis for supplying coal to a number of agencies being studied and recommendations made for award.

Cooperative work with the Air Preheater Corp., of Wellsville, N. Y., on the prevention of deposits and corrosion on air preheaters was continued, studies being included on the use of alkaline sprays, which appear to offer promising possibilities. A section entitled, "Typical Economic Study,"[58] was prepared for the chapter on "Combustion and Fuels" of the twelfth edition of Kent's Mechanical Engineers' Handbook. At the request of various Government agencies, operating and performance data were collected on a new type of automatic heating equipment using anthracite to provide information necessary for determining the economic application of such equipment to Government buildings.

Boiler Feed-Water Conditioning

Analyses and resulting recommendations were made on 9,335 samples of boiler water during the fiscal year, as follows: 3,597 from the Department of the Army; 1,973 from the Department of the Air Force; 1,692 from the Veterans Administration; 476 from the District of Columbia Government; 445 from the Department of the Navy; 276 from the Public Housing Authority; 273 from the Post Office Department; 225 from the Department of Justice; 185 from the Bureau of Indian Affairs; 83 from the Federal Security Agency; 52 from the Public Health Service; 33 from the General Services Administration; 12 from the Department of Agriculture; 10 from the Department of Commerce; and 3 from the Bureau of Mines. This is about 7-percent increase over the previous year.

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[56] Barkley, J. F., and Morgan, R. E., Fuel-Burning Equipment Dimensions Required by Smoke-Abatement Ordinances: Bureau of Mines Inf. Circ. 7557, 1950, 19 pp.

[57] Berk, A. A., and Burdick, L. R., A Method of Test for SO_2 and SO_3 in Flue Gases: Bureau of Mines Rept. of Investigations 4618, 1950, 9 pp.

[58] Morgan, R. E., Comparison of Fuels. 5. Typical Economic Study: Kent's Mechanical Engineers' Handbook Power vol., 12th ed., 1950, pp. 2-16 and 2-17, John Wiley & Sons, Inc., New York, N. Y.

Reports and recommendations covering 28 analyses of various scales, sludges, and deposits, 26 miscellaneous special analyses, and 5 analyses of boiler compounds were made.

Five hundred and eighty special Bureau of Mines field water-test kits, 10,259 bottles of chemical reagents, and 12,060 test-kit replacement items were distributed to various Government activities. Distributing these kits and reagents from a central source saved Government agencies about $15,000 this year as compared to open-market purchase.

Consulting service was given the Department of the Navy on the use of special boiler compounds in the Eleventh Naval District and on feed-water treatment of the effluent from hot process lime-soda softeners; the Department of the Air Force on condensate treatment with pressure-jet equipment at the Memphis, Tenn., airport; the Veterans' Administration on feeding phosphate to avoid feed-line deposition at Perry Point, Md., and on methods of dissolving tannin at Castle Point, Md.; the Bureau of Indian Affairs on the use of various boiler water test kits; St. Elizabeth's Hospital, Washington, D. C., on control of leakage at handhole gaskets; the Public Schools of the District of Columbia on replacing soda ash with caustic soda to minimize return-line corrosion; the Bureau of Federal Supply on use of tannin for boiler feed-water treatment; the Garnet Patterson Junior High School and the Banneker Junior High School on proper operating methods to prevent pitting; the U. S. Naval Observatory, Washington, D. C., on instructions on boiler-water sampling and testing; the U. S. Engineer Office, Philadelphia, Pa., on preparation of a manual on boiler-water treatment; and the Weather Bureau on the prevention of deposits in Alaskan plants.

At the request of the Bureau of Yards and Docks, Department of the Navy, a test of several months' duration was made on an electrolytic scheme of preventing scale in boilers at the Naval Air Station, Anacostia, D. C.; the method proved to be unsatisfactory. At the request of the Public Works Officer of the Naval Research Laboratory, Anacostia, D. C., a performance test was run on a deaerating heater.

Report was received from the Albuquerque Indian School, Albuquerque, N. Mex., that the cost of operating its boiler plant was reduced $13,700 annually by the application of Bureau of Mines boiler-water treatment practices. Based on the number of boilers used, this saving agreed closely with a previously reported saving of $139,000 annually by the Atomic Energy Commission, Los Alamos, N. Mex.; if similar savings are assumed for the thousands of Federal boilers now being served by the Bureau of Mines, some $20,000,000 per year savings are indicated.

Instruction booklets[59] for boiler-plant engineers were completed on the use of an improved tannin colorimeter for testing boiler water, on testing water for chloride content, on testing boiler water for phosphate, on testing boiler water for causticity, on the use of a hydrometer to control boiler blow-down, on testing of treated condensate for pH, on boiler-water treatment services furnished by the Boiler-Water Service Section, and on a new and improved method of testing the pH of boiler water by indicator paper.

Boiler-Water Research

Research work was continued on the use of chemicals to prevent corrosion in condensate lines in heating systems. This condensate, formed when steam condenses, is ordinarily acidic and attacks pipe vigorously; it also usually carries oxygen, which is active in corroding pipe. Satisfactory abatement of corrosion has been found by the use of several different volatile, alkaline organic chemicals called "amines." For any given plant, the cost of such treating chemicals must be balanced against the costs of maintenance and replacement of pipe. Proper choice of amines also has a bearing on over-all costs of treatment. Plants with low feed-water make-up may find it economical to use amines, and other plants, such as those having very high make-up, may find it uneconomical. For example, in three large housing developments containing more than 4,000 dwelling units, the cost of the amines ranged from 40 to 88 cents per million pounds of steam delivered, and the total cost for the entire heating season in all three plants was only $291.00. This low cost of treatment is very marked, when compared to replacement costs of more than $100,000 caused by corrosion in a few years in a similar housing development put into operation at approximately the same time but using no preventive methods. For plants where the feed-water make-up is so high that chemical treatment with amines is uneconomical, a cheaper chemical is needed; considerable progress has been made with tests to determine the conditions under which relatively inexpensive chemicals, such as ammonia, can be used successfully.

Many industrial processes require the heating or boiling of caustic soda (sodium hydroxide) solutions. All too frequently the vessels involved develop cracks in highly stressed parts, such as rivet heads, riveted seams, welds, and pipe connections. These cracks are for the most part intercrystalline and are identical to those produced in the seams of steam boilers. The results of a large number of laboratory tests are given in figure 9, which shows temperature plotted against caustic concentration.[60] All the cracking occurred in a well-defined zone, roughly above 180° F. and between 15 and 40 percent sodium hydroxide. Within this zone the probability of crack development was about 40 percent. Avoidance of the severe-cracking zone outlined by this study should greatly reduce the probability of such cracking in industrial practice, but it cannot be said that conditions outside this zone will not produce cracking in highly stressed steel over long periods of time.

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[59] a/ Instructions for Use of Improved Comparator for Tannin by Boiler-Water Color: Bureau of Mines Form BWS 9A, 1949, 4 pp.
b/ Instructions for Determining Chloride Concentration in Water by Use of Bureau of Mines Chemical Reagents: Bureau of Mines Form BWS 14, 1950, 7 pp.
c/ Instructions for Bureau of Mines Boiler-Water Test Kit for Phosphate: Bureau of Mines Form BWS 15, 1949, 7 pp.
d/ Instructions for Bureau of Mines Boiler-Water Test Kit for Causticity: Bureau of Mines Form BWS 16, 1949, 12 pp.
e/ Instructions for Using a Boiler-Water Hydrometer to Test for Density as a Guide for Blowdown Control: Bureau of Mines Form BWS 17, 1949, 7 pp.
f/ Instructions for Bureau of Mines Test Kit for pH of Treated Condensate: Bureau of Mines Form BWS 18, 1949, 3 pp.
g/ Boiler-Water Treatment Services Furnished by the Boiler-Water Service Section: Bureau of Mines Form BWS 19, 1950, 23 pp.
h/ Instructions for Test Kit for pH of Boiler Water by Indicator Paper: Bureau of Mines Form BWS 20, 1950, 3 pp.

Smoke Abatement

Work on smoke-abatement problems continued quite active throughout the United States. Cities requesting Bureau of Mines publications and consulting service include the following: Providence, R. I.; Homewood, Ill.; Dearborn and Wyandotte, Mich.; East Syracuse, Great Neck, and Penn Yan, N. Y.; Cumberland, Md.; Indianapolis, Ind.; Bogota, N. J.; Springfield and Dayton, Ohio; Des Moines, Iowa; and Philadelphia, Pa. Upon request, an engineer of the Bureau of Mines met in conference with the smoke-regulation engineers of a number of cities in the Detroit-Windsor area to discuss proper handling of smoke-abatement problems. Work on smoke-abatement problems for the International Joint Commission was continued by its Technical Advisory Board on Air Pollution, which includes a Bureau of Mines engineer. Recommendations were made for abating the smoke from vessels on the Detroit River and its tributaries, including the establishment of a jurisdictional authority and the wording of a smoke-abatement ordinance.

Combustion

A chapter on combustion of solid fuels was published in the twelfth edition of Kent's Mechanical Engineers' Handbook.[61] This chapter discussed principles of combustion, stoichiometry and energetics of combustion, and combustion calculations of two types of material balances and an energy or heat-balance type.

Corrosion of Wall Tubes in Steam Generators

Extended studies of the mechanism of external corrosion of furnace-wall tubes of high-capacity, slag-tap boiler furnaces were begun in 1942 by the Bureau of Mines in cooperation with the Combustion Engineering Co. These studies have shown that liquid alkali-metal pyrosulfates can be formed under operating conditions from deposits of alkali-metal sulfates on the tubes. The rate of attack will be considerably higher when the alkali-metal sulfates, or "enamel" deposits, form a liquid phase than when a liquid phase is not formed. The phase boundaries for three compositions of alkali-metal sulfates in the system M_2SO_4-SO_3 have been established. The conditions for the thermal decomposition of coal ash, leading to the formation of the SO_3 necessary for the corrosion process, have been determined. Alkali-metal sulfate deposits on furnace tubes are believed to result from volatilization of alkalies in the coal, which condense as corresponding oxides on the tubes and then convert to sulfates by reaction with SO_3 in the furnace atmosphere. Furnace-wall-tube attack, associated with deposits consisting mainly of FeS, is believed to be related to the deposition of pyrites on the tubes. The pyrites originate from coarse coal or coal that is poorly distributed in the burners with respect to size. It adheres to deposits, previously formed, of alkali-metal sulfates, oxidizes relatively slowly to

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[60] Berk, A. A., and Waldeck, W. F., Caustic Danger Zone: Chemical Eng., vol. 57, June 1950, pp. 235, 236, 238.

[61] Corey, Richard C., and Graf, Ernst G., Combustion: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-03 to 2-12, John Wiley & Sons, Inc., New York, N. Y.

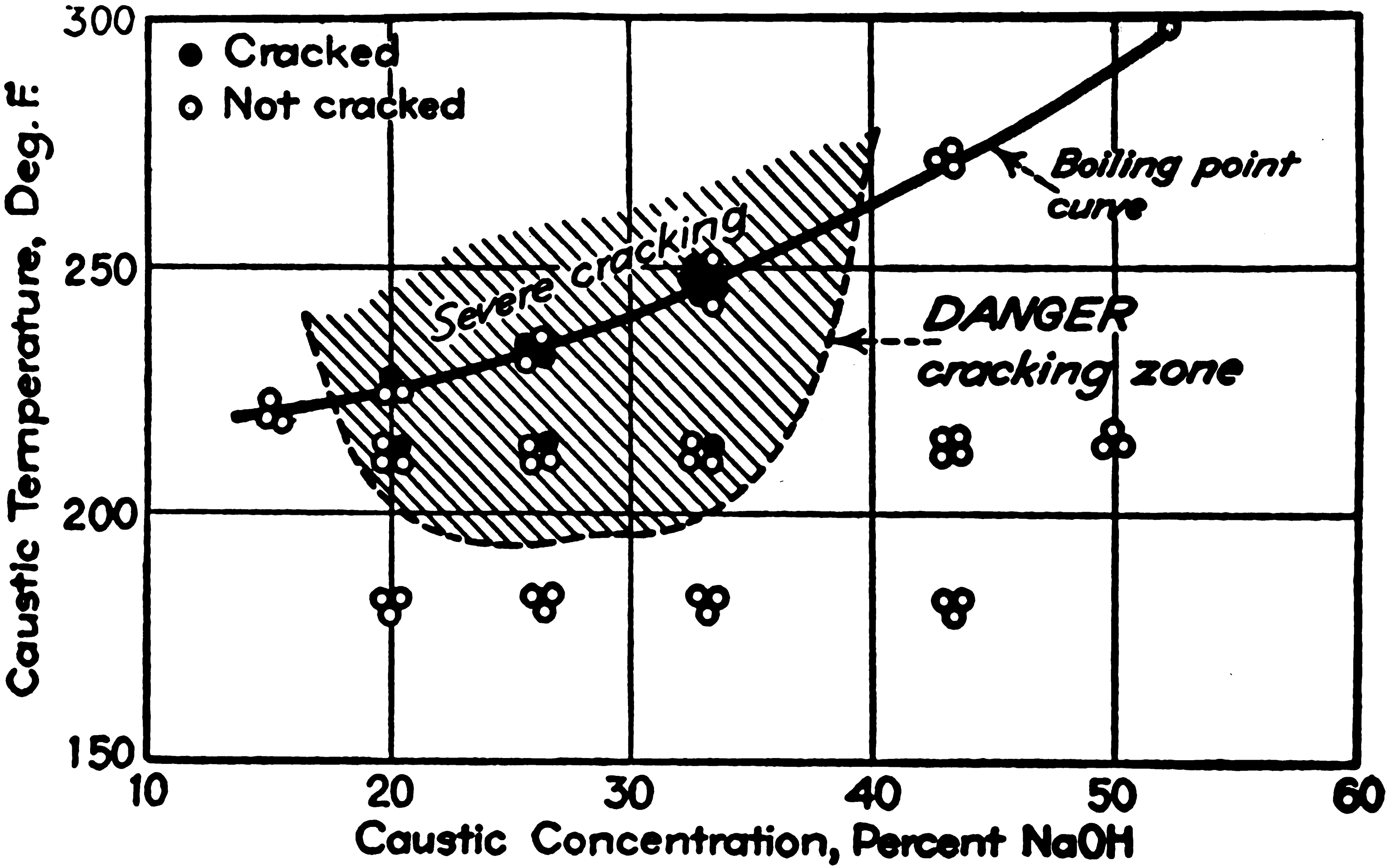

Figure 9. - Conditions of temperature and concentration of sodium hydroxide under which cracking of steel container may readily occur.

FeS and Fe_3O_4, and thereby produces sufficient SO_3 to react with the alkali-metal sulfates and the oxide on the metal. The net effect is to cause corrosion by the same processes that have been ascribed to corrosion by the alkali-metal-sulfate type of deposit. The relationship between the results obtained in the laboratory and observations of furnaces before and after preventive measures have been applied is described.62/

Factors Affecting Heat Absorption in Furnaces

As part of the general program of investigations by the A.S.M.E. Special Research Committee on Furnace Performance Factors, the Bureau is making a series of determinations of furnace-heat absorption in large-utility power boiler furnaces. During the course of these studies, modifications have been made in the test technique to improve the precision of furnace testing. Automatic recording instruments have been employed, which permit more detailed sampling and examination of furnace-outlet gases than heretofore, without an appreciable increase in the time or work required. Improved designs of radiation shields have been developed for high-velocity thermocouples, with accuracy comparable to the B & W multiple-shield, high-velocity thermocouple, but which may be employed conveniently in pulverized-coal-fired furnaces. Detailed descriptions of the distribution of gas composition and temperature at the outlet of a 640,000-pound-per-hour horizontally fired boiler furnace have been published.63/ Based upon this knowledge, the sampling characteristics of the gases at the furnace outlet for furnace heat-absorption measurements have been examined, and a procedure is suggested for the rational selection of simplified sampling schedules. The methods of calculating furnace-heat absorption from the temperature and gas-composition data are examined critically, placing special emphasis on the effect of weighting with respect to mass velocity. Suggestions are presented whereby the significance of mass velocity for furnace heat-absorption calculations may be determined from the temperature and gas-composition data.

Steam-Dried Lignite

A large-scale combustion test using steam-dried lignite was carried out at the Otter Tail Power Co. plant at Crookston, Minn. The unit used was a 5,000-kw. pulverized-coal unit. Table 5 shows the pertinent results. These results confirm former tests and show improved efficiency through drying.

TABLE 5. - Combustion of pulverized lignite, Otter Tail Power Co., Crookston, Minn.

Type of lignite	Kincaid	Dakota Star
Condition of lignite	Natural	Steam-dried
Moisture ... percent	32.5	15.6
Heating value as fired ... B.t.u./lb.	7,270	9,170
Heat input/kw.-hr. generated ... B.t.u.	14,902	14,481
Lignite consumed as fired ... lb./kw.-hr.	2.05	1.58
Heat absorbed by water and steam in the boiler of total heat input ... percent	76.4	81.5

62/ Corey, R. C., Grabowski, H. A., and Cross, B. J., External Corrosion of Furnace-Wall Tubes. III. Further Data on Sulphate Deposits and the Significance of Iron Sulphide Deposits: Trans. Am. Soc. Mech. Eng., vol. 71, November 1949, pp. 951-963.

63/ Cohen, P., Corey, R. C., and Myers, J. W., Methods and Instrumentation for Furnace Heat-Absorption Studies: Temperature and Composition of Gases at Furnace Outlet: Trans. Am. Soc. Mech. Eng., vol. 71, November 1949, pp. 965-978.

CARBONIZATION OF COAL

Coke Production

Our fuel reserves were reviewed,[64] with special reference to coking coals, and the problem of utilizing progressively poorer coking coals as reserves of high-quality coals approach depletion was discussed.

Methods of meeting shortages of metallurgical coke were described to the United Nations Scientific Conference on the Conservation and Utilization of Resources held at Lake Success, N. Y., in August 1949.[65] Among the methods that relieved shortages of coke in the United States during World War II were: Reconditioning and putting into service all available slot-type and beehive ovens, since enough new ones cannot be built in time; acceleration of operations by increased temperatures and earlier pushing of coke; curtailing of nonessential uses; and improving uniformity and quality of coke by better cleaning, blending, and mixing and higher bulk density.

The threat of continuing shortages resulting from the depletion of premium grades of coking coal may be met by increased exploration and by conservation through limitations on the use of coking coal for other than coke production and by the reduction of mining losses. Conservation is aided by improved cleaning methods, which permit lower grades to replace premium grades of coal, and by blending that broadens the range of metallurgical coals.

New methods of blast-furnace operation and gas production give promise of reducing coke requirements, and there are prospects of developing coke-free methods of iron-ore reduction. Continued research is essential to develop ovens capable of coking a broader range of coals.

Survey of Carbonizing Properties of American Coals

The scope of the survey of the carbonizing properties of American coals remained the same as in the previous year, when it was narrowed by limiting experimental carbonization to tests at high temperatures. Emphasis continued to be placed upon research bearing directly on the manufacture of metallurgical coke. A high proportion of the tests were made on blends because most metallurgical coke is made from mixed coals; furthermore, the carbonizing properties of some coals coked singly are of little value in predicting the properties of cokes made from their blends.

Forty-six coals were tested during the year, and blends of most of these were carbonized. The source of coals and composition of the blends tested (carbonization, plasticity, oxidation, or expansion tests) are given in table 6. Proximate and ultimate analyses of the coals, physical properties of coke, and a summary of yield data have been published.[66]

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[64] Fieldner, Arno C., Coal for Coke Production: Bureau of Mines Inf. Circ. 7559, 1950, 21 pp.

[65] Fieldner, A. C., and Newman, L. L., Overcoming Shortages of Metallurgical Coke: Mech. World and Eng. Rec., vol. 126, No. 3272, Sept. 30, 1949, pp. 402-405.

[66] Davis, J. D., and Reynolds, D. A., Survey of Carbonizing Properties of American Coals - Bureau of Mines Coal Carbonization Laboratory, 1948-49: Proc. Am. Gas Assoc., 1949, pp. 413-424.

TABLE 6. - Description of coals and blends

Coal No.	Description
	Kentucky
381	Elkhorn No. 1 bed, Elk Horn No. 28 mine, Floyd County, Ky.
a381	Elkhorn No. 1 bed, Elk Horn No. 28 mine, Floyd County, Ky. (washed).
381A	Blend: 80 percent Elkhorn No. 1 (381) and 20 percent Pocahontas No. 3 (f75).
381B	Blend: 70 percent Elkhorn No. 1 (381) and 30 percent Pocahontas No. 3 (f75).
382	Elkhorn No. 2 bed, Turner No. 5 mine, Floyd County, Ky.
382A	Blend: 80 percent Elkhorn No. 2 (382) and 20 percent Pocahontas No. 3 (f75).
382B	Blend: 70 percent Elkhorn No. 2 (382) and 30 percent Pocahontas No. 3 (f75).
383	Leatherwood bed, Leatherwood mine, Perry County, Ky.
383A	Blend: 80 percent Leatherwood (383) and 20 percent Pocahontas No. 3 (f75).
383B	Blend: 70 percent Leatherwood (383) and 30 percent Pocahontas No. 3 (f75).
384	Harlan bed, Path Fork mine, Harlan County, Ky.
384A	Blend: 80 percent Harlan (384) and 20 percent Pocahontas No. 3 (f75).
384B	Blend: 70 percent Harlan (384) and 30 percent Pocahontas No. 3 (f75).
	West Virginia
345	Davy-Sewell bed, Twin Branch mine, McDowell County, W. Va.
a368	Pittsburgh bed, Jamison No. 9 mine, Marion County, W. Va.
a368A	Blend: 80 percent Pittsburgh (a368) and 20 percent Pocahontas No. 3 (g75).
a368B	Blend: 70 percent Pittsburgh (a368) and 30 percent Pocahontas No. 3 (g75).
378	Firecreek bed, Laurel Creek mine, Greenbrier County, W. Va.
379	Firecreek bed, Dunedin No. 1 mine, Raleigh County, W. Va.
379A	Blend: 20 percent Firecreek (379) and 80 percent Pittsburgh (p28).
379B	Blend: 30 percent Firecreek (379) and 70 percent Pittsburgh (p28).
406	Sewell bed, Williams River mine, Webster County, W. Va.
407	Eagle bed, Cannellton No. 3 mine, Fayette County, W. Va.
413	Upper Freeport bed, Bull Run No. 1 mine, Preston County, W. Va.
413A	Blend: 20 percent Upper Freeport (413) and 80 percent Pittsburgh (q28).
413B	Blend: 30 percent Upper Freeport (413) and 70 percent Pittsburgh (q28).
413C	Blend: 50 percent Upper Freeport (413) and 50 percent Pittsburgh (q28).
425	Sewell bed, Duo mine, Greenbrier County, W. Va.
425A	Blend: 20 percent Sewell (425) and 80 percent Pittsburgh (r28).
425B	Blend: 30 percent Sewell (425) and 70 percent Pittsburgh (r28).
426	Sewell bed, Bergoo No. 6 mine, Webster County, W. Va.
426A	Blend: 80 percent Sewell (426) and 20 percent Pocahontas No. 3 (g75).
427	Sewell bed, Williams-River mine, Webster County, W. Va.
427A	Blend: 80 percent Sewell (427) and 20 percent Pocahontas No. 3 (g75).
428	Sewell bed, Sterling-Sewell mine, Nicholas County, W. Va.
428A	Blend: 70 percent Sewell (428) and 30 percent Pittsburgh (r28).

TABLE 6. - Description of coals and blends - Continued

Coal No.	Description
	West Virginia - Continued
429	Sewell bed, Cassity No. 2 mine, Randolph County, W. Va.
429A	Blend: 80 percent Sewell (429) and 20 percent Pittsburgh (r28).
429B	Blend: 70 percent Sewell (429) and 30 percent Pittsburgh (r28).
429C	Blend: 50 percent Sewell (429) and 50 percent Pittsburgh (r28).
	British Columbia - Alaska - Western
385	No. 3 bed, Evans-Jones mine, Lower Matanuska Valley, Alaska.
385A	Blend: 70 percent No. 3 (385) and 30 percent "M" or "N" bed (386).
386	"M" or "N" bed, Chickaloon mine, Upper Matanuska Valley, Alaska.
388	No. 8 mine, Comox Lake district, Vancouver Island, British Columbia.
389	T'Sable River mine, Nelson district, Vancouver Island, British Columbia.
390	No. 9 bed, Elk River No. 9 mine, Fort Steele district, British Columbia.
391	No. 10 bed, Elk River No. 1 mine, Fort Steele district, British Columbia.
392	No. 3 bed, Elk River No. 3 mine, Fort Steele district, British Columbia.
393	No. 4 bed, Elk River No. 4 mine, Fort Steele district, British Columbia.
394	Michel mine (raw coal), Fort Steele district, British Columbia.
395	Michel mine, Fort Steele district, British Columbia.
398	No. 5 bed, Roslyn No. 3 mine, Kittitas County, Wash.
399	Peace River district, Hudson's Hope, British Columbia.
400	Robertson mine, Queen Charlotte Island, British Columbia.
401	Aldridge Creek prospect, Elk River No. 5 mine, Fort Steele district, British Columbia.
402	Aldridge Creek prospect, Elk River No. 4 mine, Fort Steele district, British Columbia.
423	Lower Sunnyside bed, Horse Canyon mine, Carbon County, Utah.
423A	Blend: 82 percent Lower Sunnyside (423), 15 percent Elk River No. 4 (393), and 3 percent pitch.
423B	Blend: 82 percent Lower Sunnyside (423), 15 percent Elk River No. 1 (391), and 3 percent pitch.
423C	Blend: 82 percent Lower Sunnyside (423), 15 percent Elk River No. 9 (390), and 3 percent pitch.
423D	Blend: 85 percent Lower Sunnyside (423) and 15 percent Pocahontas No. 3 (g75).
423E	Blend: 97 percent Lower Sunnyside (423) and 3 percent pitch.
423F	Blend: 85 percent Lower Sunnyside (423) and 15 percent Oklahoma low-volatile (424).
423G	Blend: 82 percent Lower Sunnyside (423), 15 percent Oklahoma low-volatile (424), and 3 percent pitch.
424	Oklahoma (Republic) low-volatile coal (Geneva Steel Co.).
	South America
423	No. 1 bed, Cerejon region, Colombia, South America.
433	No. 2 bed, Cerejon region, Colombia, South America.

TABLE 6. - Description of coals and blends - Continued

Coal No.	Description
	Miscellaneous
346	Upper and Lower Freeport beds, Kent Nos. 1 and 2 mines, Indiana County, Pa.
380	Lower Freeport bed, Kent No. 1 mine, Indiana County, Pa.
380A	Blend: 50 percent Lower Freeport (380) and 50 percent Upper Freeport (387).
380B	Blend: 33-1/3 percent Lower Freeport (380) and 66-2/3 percent Upper Freeport (387).
387	Upper Freeport bed, Kent No. 2 mine, Indiana County, Pa.
397	Upper Freeport bed, Kent No. 7 mine, Indiana County, Pa.
414	Elnora (No. 4) bed, Maid Marian (strip) mine, Daviess County, Ind.
415	Illinois No. 6 bed (raw coal).
416	Jones & Laughlin Steel Corp. C-Battery Mix (G-72 or P-45): 10 percent low-volatile Pocahontas No. 6; 40 percent high-volatile A "Dixie" (mixture of coals from Kentucky, West Virginia, and Tennessee); and 50 percent high-volatile A washed Vesta.
417	High-volatile A washed Shannopin coal (G-35 or P-27) from J. & L. mine: 1/4-inch float 1.35.
417A	90 percent coal 417 and 10 percent 535° C. coke made from coal 417: 1/4-inch float 1.55.
430	Pittsburgh bed, Champion No. 1 mine (feed coal).
431	Pittsburgh bed, Champion No. 1 mine (face discharge refuse).
	Blending coals
p28	Pittsburgh bed, Warden mine, Allegheny County, Pa.
q28	
r28	
f75	Pocahontas No. 3 bed, Kimball, McDowell County, W. Va.
g75	
f75A	Blend: 80 percent Pittsburgh (p28) and 20 percent Pocahontas No. 3 (f75).
f75B	Blend: 70 percent Pittsburgh (p28) and 30 percent Pocahontas No. 3 (f75).

Methods of Testing

The carbonizing properties of the eastern coking coals were determined by Bureau of Mines-American Gas Association (BM-AGA) tests at 800° and 900° C. in the standard 18-inch retort. One coal and five blends were also carbonized in the 500-pound slot oven. Physical properties of the cokes from these tests were determined by standard methods of the American Society for Testing Materials.

The carbonizing properties of the British Columbian, Alaskan, Western, and Colombian coals were determined by BM-AGA tests at 900° C. in the standard 13-inch retort. These cokes were tested by BM-AGA methods; the tumbler test is less severe than the A.S.T.M. method.

Coals Tested

A relatively large proportion of the carbonization work was done on samples representing the Elkhorn No. 1, Elkhorn No. 2, Leatherwood, and Harlan beds in Kentucky and the Fire Creek, Upper Freeport, Pittsburgh, and Sewell beds in West Virginia. Five Sewell coals from four counties in West Virginia were tested because this bed has large reserves and its rank ranges from low to high volatile. The carbonizing properties of 13 British Columbian and two Alaskan coals were determined in a cooperative investigation with the United States Steel Co., which was conducted to determine the feasibility of substituting coal from the northern part of this continent for low-volatile Oklahoma coal now blended with Lower Sunnyside coal for the production of metallurgical coke in Utah. The Alaskan coals were from the upper and lower Matanuska Valley; the British Columbian coals included two from Vancouver Island, seven from Elk River beds Nos. 3, 4, 5, 9, and 10 in the Fort Steele district, two Michel-mine coals, one from the Peace River district, and one from the Queen Charlotte Isles. Lower Sunnyside coal from the Horse Canyon mine, Carbon County, Utah, was blended with the most strongly coking British Columbia coals. Two samples from Magdalena, Colombia, South America, were carbonized singly.

Sewell-Bed Coal

The Sewell coal bed extends from the southern boundary of West Virginia northeast to Preston County, which borders Pennsylvania, and its original tonnage was estimated to be about 6-1/4 billion tons. The coal rank ranges from low-volatile in the south to high-volatile A in the north. Because only a few high-ranking low-volatile coals from the southern part of this bed had been tested previously, the survey was extended to those counties containing medium- and high-volatile coal. The variation of carbonizing and expanding properties of Sewell bed with rank and geographical location is shown in table 7. The following five coals were tested: (1) Duo mine in Greenbrier County (425), Bergoo No. 6 mine in Webster County (426), Williams River mine in Webster County (427), Sterling-Sewell mine in Nicholas County (428), and Cassity No. 2 mine in Randolph County (429). These coals ranked either low in the medium-volatile group or high in the high-volatile A group; they contained 67.0 to 72.5 percent fixed carbon on the dry, mineral-matter-free basis. Their contents of ash and sulfur as carbonized (3.1 to 8.0 and 0.6 to 1.1 percent, respectively) showed that they qualified chemically for the manufacture of metallurgical coke. The ranges in strength indexes of the 900° C. cokes were: 1-1/2-inch shatter, 85-90; 1-inch tumbler, 43-50; and 1/4-inch tumbler, 59-66. The experimental data indicate that these coals are suitable for commercial carbonization and that they could be blended with coals of different rank. Duo-mine coal should be blended with high-volatile A coal to lower its expansion and thereby avoid damage to oven walls. Bergoo No. 6, Sterling-Sewell, and Cassity No. 2 coals coked strongly when carbonized singly; possibly, they could be carbonized without blending if high-charge densities were avoided. Williams River coke had lower tumbler indexes than the others, and because these indexes were raised appreciably by blending with 20 percent Pocahontas No. 3, blending with low-volatile coal is recommended for commercial carbonization.

TABLE 7. - Survey of carbonizing properties of Sewell-bed coal from West Virginia

Coal No.	Mine	County	Dry, mineral-matter-free, fixed carbon, percent	As received, percent			Coke index, percent		Expansion, percent
				Volatile matter	Ash	Sulfur	Shatter, 1-1/2-inch screen	Tumbler, 1-inch screen	
26..	Cranberry	Raleigh...	78.6	20.5	2.1	0.8	86.9	[1]69.6(58.6)	-
55..	Wyoming	Wyoming...	77.5	22.0	2.9	.7	85.7	[1]68.9(58.9)	+16.0
27..	Summerlee	Fayette...	72.5	26.5	2.4	.5	79.4	[1]68.0(58.0)	-
425.	Duo	Greenbrier	72.5	26.2	3.1	1.1	90.0	54.0	+23.1
426.	Bergoo	Webster ..	69.5	28.6	5.4	.6	85.0	50.0	-11.7
429.	Cassity	Randolph..	69.2	28.3	7.4	.6	96.0	58.0	- 4.7
427.	Williams-River.	Webster ..	67.4	30.5	5.5	.6	90.0	43.0	- 7.0
428.	Sterling-Sewell	Nicholas..	67.0	30.9	4.3	.6	86.9	54.2	- 1.5
	Average		71.8	26.7	4.1	.7	87.5	58.2	-
	High		78.6	30.9	7.4	1.1	96.0	69.6	-
	Low		67.0	20.8	2.1	.5	79.4	43.0	-

[1] Figures in parentheses are estimated for A.S.T.M. tumbler; the BM-AGA 1-inch index (used in early work) is about 10 points higher than the A.S.T.M.

British Columbia, Alaska, and Western Coals

The coals from Upper and Lower Matanuska Valley in Alaska differed markedly in coking power. No. 3-bed coal (385) yielded 63.2 percent char, which was poorly fused and too weak to test. M-bed coal (386) expanded strongly and yielded 78.2 percent coke, which was very strong. The latter should be valuable for blending with lower-rank (high-volatile) coals; it contained 12.7 percent ash and 0.5 percent sulfur.

Both samples from Vancouver Island ranked as high-volatile A and coked strongly. No. 8-mine coal (388) yielded 69.5 percent coke, and T'Sable River-mine coal (389) yielded 71.3 percent. The indexes of the respective cokes were: 1-1/2-inch shatter, 82 and 77; 1-inch tumbler, 56 and 59; and 1/4-inch tumbler, 65 and 65. These indexes are higher than the average for coals of similar rank.

Lower Banner Coal, Russell County, Va.

The carbonizing properties of Lower Banner-bed coal from No. 56 mine, Dante, Russell County, Va., were determined by BM-AGA tests at low, medium, and high temperatures and by expansion tests in the sole-heated oven.[67] This coal ranked as high-volatile A bituminous, and its yields of carbonization products approximated the average yields for coals of similar rank. The cokes made at all temperatures were well-fused; the high-temperature cokes were medium-grained and moderately fissured. The 900° C. coke resisted breakage by impact well but was more abradable than the average BM-AGA coke made from high-volatile A coals. Blending with Pocahontas No. 3 coal strengthened the 900° C. coke materially, and the blend containing 30 percent of this low-volatile coal coked more strongly than the blend containing 20 percent. Three samples from mine No. 56 contracted 22.2 to 25.8 percent in the sole-heated oven at the standard bulk density of 55.5 pounds per cubic foot. The blends containing 20 and 30 percent Pocahontas No. 3 contracted 11.5 and 6.0 percent, respectively. Oxidation tests in air at 100° C. for periods of 3.9, 11.2, and 19.2 days showed that Lower Banner oxidized more rapidly than Pittsburgh coal.

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[67] Davis, J. D., Reynolds, D. A., Brewer, R. E., Ode, W. H., Naugle, B. W., and Wolfson, D. E., Carbonizing Properties of Lower Banner Coal from No. 56 Mine, Dante, Russell County, Va.: Bureau of Mines Tech. Paper 720, 1949, 45 pp.

Thick Freeport and Pittsburgh Coals, Pennsylvania; Elkhorn Coal, Kentucky; and America and Mary Lee Coals, Alabama

The carbonizing properties of nine coals mined and carbonized commercially by the Republic Steel Corp. were determined in a cooperative investigation with that firm.[68] BM-AGA carbonization tests were made at 800°, 900°, and 1,000° C. on the single coals because the corporation was particularly interested in the yields of carbonization products. Two Thick Freeport-bed coals represented the Indianola and Russellton mines in Allegheny County, Pa.; two Pittsburgh coals represented the Clyde and Crescent mines in Washington County, Pa.; Elkhorn-bed coal was from the Republic mine, Pike County, Ky.; America-bed coal was obtained from the Virginia mine, Jefferson County, Ala.; and two Mary Lee-bed coals were from Sayreton mines Nos. 1 and 2 and Sayre mine, Jefferson County, Ala. The Alabama coals ranked higher and yielded more coke and less other products than the other coals. Cokes from the Thick Freeport, Pittsburgh, and Elkhorn coals were well-fused, medium-grained, and moderately fissured; America and Mary Lee cokes were finer-grained and less-fissured. The heating values of the gases in B.t.u. per pound of coal were: Thick Freeport, Indianola and Russellton mines, 3,180 and 3,120, respectively; Pittsburgh, Crescent and Clyde mines, 3,020 and 3,060, respectively; America, 2,700; Mary Lee, Sayreton mines Nos. 1 and 2, 2,760, Sayre mine, 2,750, and Sayreton No. 2 mine, 2,870. The expansions (or contractions) in the sole-heated oven at 55.5 pounds per cubic foot were: Thick Freeport, Indianola mine, -12.7, and Russellton mine, -14.6; Pittsburgh, Crescent mine, -16.6, and Clyde mine, -15.3; Elkhorn, -11.1; America, -4.3; and Mary Lee, Sayreton No. 2 mine, -2.9, Sayreton Nos. 1 and 2 mines, -7.3, and Sayre mine, -11.3.

Testing Alabama Coals in the 17-Inch Test Oven

Test work was continued on the carbonizing properties of Alabama coals using the 17-inch, 1/2-ton charge, experimental, movable wall coke oven at the Bureau of Mines Southern Experiment Station, Tuscaloosa, Ala. The coals tested were from the northwest and northeast portions of the Warrior field and the northern section of the Cahaba coal field. These coals covered the range of volatile matter (25 to 37 percent) into which substantially all of the Alabama coals fall. The coals were carbonized at 2,000° and 2,400° F., and the flue temperature resultant coke was evaluated in terms of the Standard A.S.T.M. tests for coke quality. The wall force exerted during carbonization was also determined. The oven has been found satisfactory for testing purposes.

Foundry Cokes from Sunnyside Coal, Utah

A study of the possibilities of making foundry coke from the lower Sunnyside seam coal of Carbon County, Utah, with blends of low-volatile coal, pitch, and medium-volatile char, was initiated at Denver in cooperation with the Utah Fuel Co.

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[68] Davis, J. D., Reynolds, D. A., Naugle, B. W., Wolfson, D. E., Birge, G. W., Flickinger, C. H., and Graham, J. P., Carbonizing Properties of Thick Freeport and Pittsburgh Coals from Pennsylvania, Elkhorn Coal from Kentucky, and America and Mary Lee Coals from Alabama. Application of BM-AGA Carbonization Test Results to Byproduct Practice: Tech. Paper 726, 1949, 58 pp.

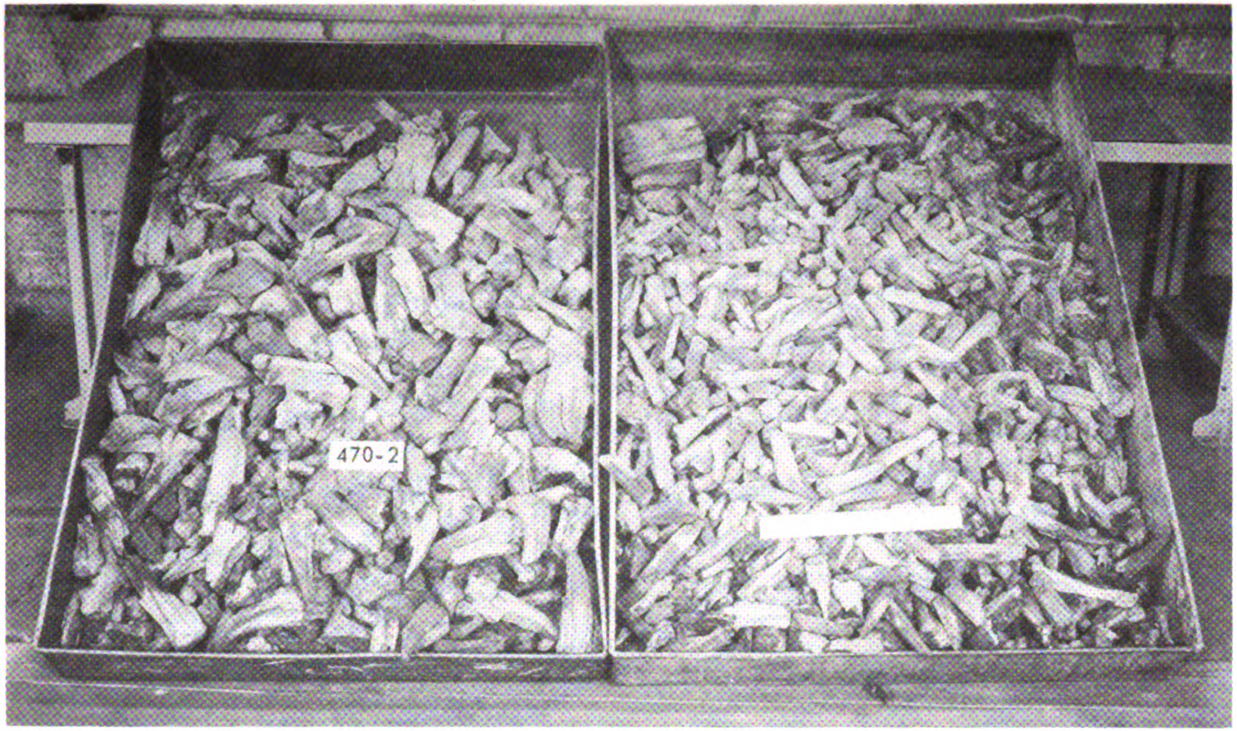

Figure 10. - Coke from 100 percent Lower Sunnyside coal, showing typical fingery structure.

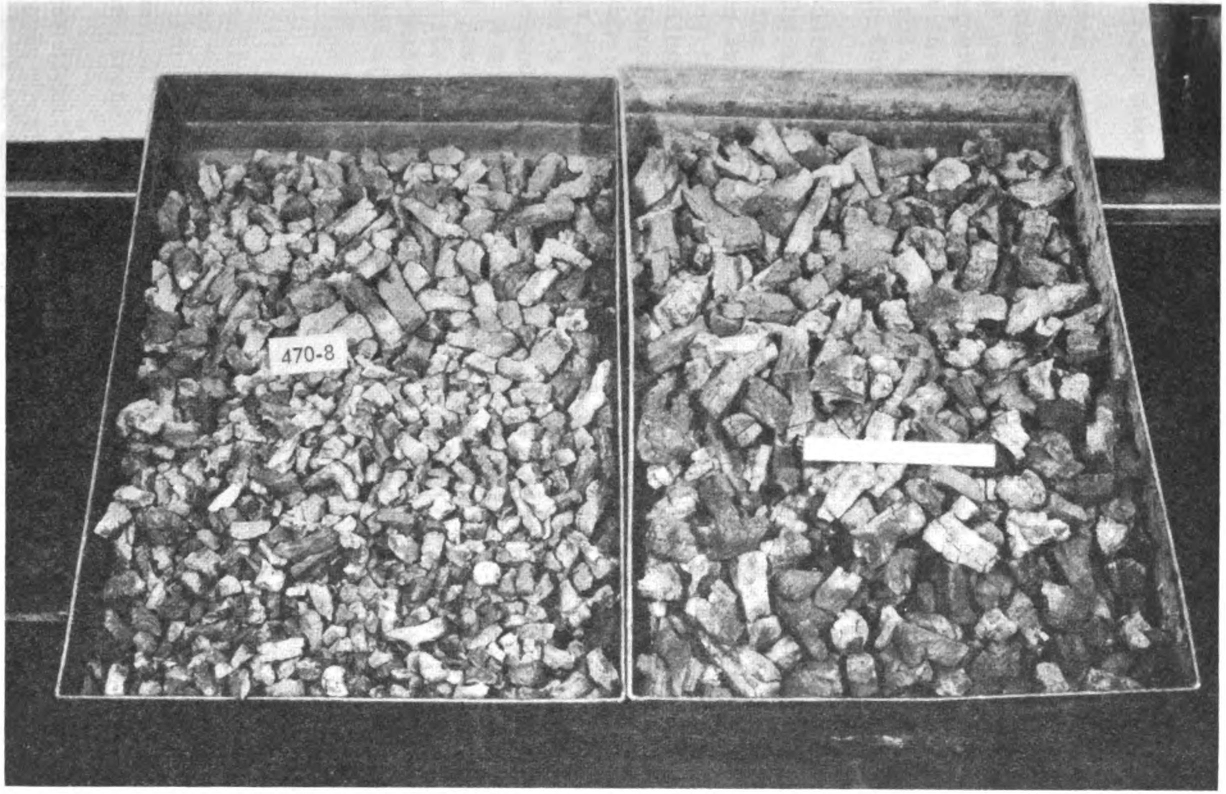

Figure 11. - Coke from 70 percent Lower Sunnyside coal plus 30 percent medium-volatile coal from Oklahoma, showing blocky structure obtained.

Blast-furnace coke is being produced from this coal in three byproduct coke plants, and foundry coke is being produced at one beehive plant. The byproduct coke made from Sunnyside coal is of poor quality when judged by eastern practice, but western blast-furnace operators dependent upon this coke have developed techniques that permit them to use this material efficiently. This coke is not suitable for foundry use. The beehive plant does not supply sufficient foundry coke to meet the demand in the western States, and the economics of the industry do not favor the erection of new beehive-coke plants. Foundry coke is currently being imported from all of the major coke-producing areas of the country.

A program specifically aimed at determining the optimum blend of Sunnyside coal with other materials that will yield a suitable foundry coke in byproduct ovens was undertaken by the Bureau in cooperation with the Utah Fuel Co., one of the major producers of metallurgical-grade Sunnyside coal. Carbonization tests were made in a 500-pound experimental slot oven under time and temperature conditions similar to those found in commercial byproduct ovens using the Sunnyside coal. The cokes produced from this oven were tested by A.S.T.M. standards and by methods and techniques developed in the Utah byproduct coke plants.

After determining the properties of coke made from straight Sunnyside coal, the effect of the addition of 5 and 10 percent of coal-tar pitch were studied, followed by the testing of 10, 20, and 30 percent blends of a low-volatile bituminous coal from western Arkansas and a 30 percent blend with a medium-volatile coal from Oklahoma. Marked improvements in the physical properties were produced by the addition of the blending materials, the most satisfactory blend tested to date being the 70 percent of Sunnyside coal with 30 percent of the medium-volatile Oklahoma coal. The results of the physical tests made on the cokes are shown in table 8. One of the most notable improvements was in the production of a blocky coke. Straight Sunnyside coal produces a fingery coke, as shown in figure 10, that is not satisfactory for use in the foundry cupolas because of its low packing density and inherently weak structure. A more blocky coke, produced from a blend of 70 percent lower Sunnyside coal and 30 percent medium-volatile coal from Oklahoma, is shown in figure 11. In addition to its improved shape, this coke is also more resistant to degradation than is the coke made from the straight Sunnyside coal. The addition of coal-tar pitch increased the average size of the coke but did not produce a coke strong enough for the purpose.

Low-temperature Carbonization of Noncoking Coals

An investigation of the low-temperature carbonization of the lower-rank coals was initiated to determine the technical and economic possibilities of carbonizing these coals for production of char for electric-power generation and tars for industrial chemicals. Pilot plants for drying and carbonizing lignite in the entrained and fluidized state were operated successfully on a scale of 2 to 10 tons per day.

When raw lignite containing 37 percent moisture is carbonized at 900° F., more than 75 percent of the available heat in the raw fuel is confined in the char, which represents about 45 percent of the weight of the original lignite; furthermore, about 15 percent of the heat in the raw lignite is recovered as tar. These high-grade products from lignite are valuable industrial raw materials, and their cost of transportation should be considerably less than the cost of shipping the same quantity of heat in the form of raw lignite. The processed materials are also more uniform and stable in physical and chemical properties than the raw fuel.

TABLE 8. - Physical properties of cokes produced from Utah Sunnyside coal and blends

Charge composition	100 percent Sunnyside	95 percent Sunnyside, 5 percent coal-tar pitch	90 percent Sunnyside, 10 percent coal-tar pitch	90 percent Sunnyside, 10 percent Arkansas low-volatile coal	80 percent Sunnyside, 20 percent Arkansas low-volatile coal	70 percent Sunnyside, 30 percent Arkansas low-volatile coal	70 percent Sunnyside, 30 percent Oklahoma medium-volatile coal
Charging density.............lb./cu.ft.	52	52	52	52	52	52	52
Flue temperature ....°F.	2,300	2,300	2,300	2,300	2,300	2,300	2,300
Carbonization time ....hours	16.5	16.5	16.5	16.5	16.5	16.5	16.5
Average inside oven wall temperature, °F.	1,859	1,879	1,882	1,849	1,869	1,904	1,873
Average coke size[1] ....inches	1.66	1.79	2.15	1.86	2.02	2.09	2.17
Drop shatter test, Columbia Steel Co. method:[2]							
Cumulative percentage retained on 1-1/2-inch screen	30.0	43.3	64.0	48.0	65.3	73.3	72.7
Tumbler test, Columbia Steel Co. method:[2]							
Cumulative percentage retained on 1.05-inch screen	43.3	68.7	76.7	73.3	84.7	86.7	84.0
Cumulative percentage retained on 0.263-inch screen	86.7	90.0	91.3	90.0	91.3	90.7	90.0
Shape factor[3]	1.16	1.04	0.88	1.00	0.68	0.76	0.67

[1] Square-hole screens.

[2] Reynolds, D. A., Davis, J. D., Brewer, R. E., Ode, W. H., Wolfson, D. E., and Birge, G. W., Carbonizing properties of Western Coals: Bureau of Mines Tech. Paper 692, 1946, 79 pp.

[3] Shape factor is a measure of the ratio of length of piece to average diameter. Fingery cokes have shape factors over 1, and satisfactory foundry cokes have shape factors of 0.60 to 0.70.

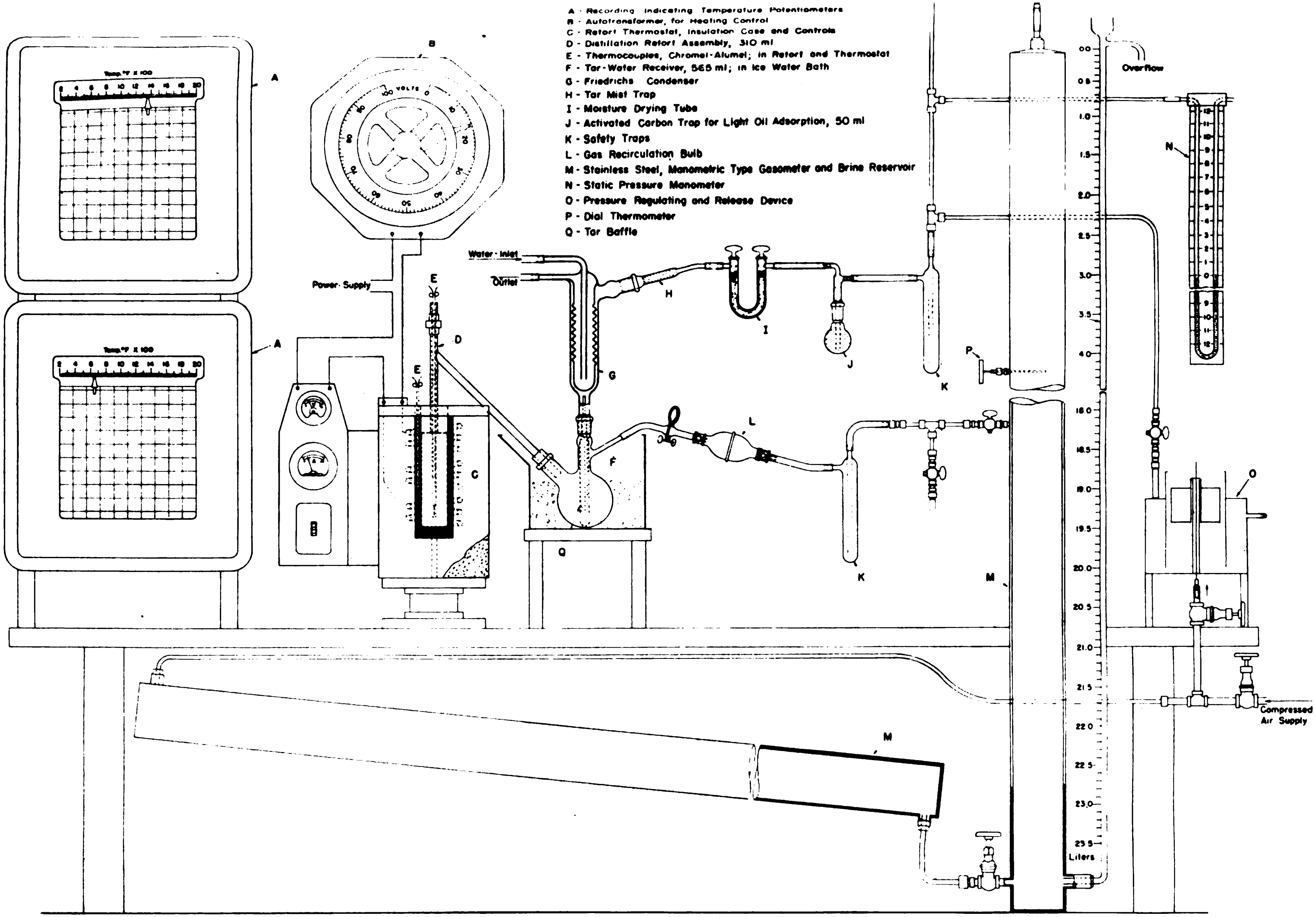
A - Recording Indicating Temperature Potentiometers
B - Autotransformer, for Heating Control
C - Retort Thermostat, Insulation Case and Controls
D - Distillation Retort Assembly, 310 ml
E - Thermocouples, Chromel-Alumel; in Retort and Thermostat
F - Tar-Water Receiver, 565 ml; in Ice Water Bath
G - Friedrichs Condenser
H - Tar Mist Trap
I - Moisture Drying Tube
J - Activated Carbon Trap for Light Oil Adsorption, 50 ml
K - Safety Traps
L - Gas Recirculation Bulb
M - Stainless Steel, Manometric Type Gasometer and Brine Reservoir
N - Static Pressure Manometer
O - Pressure Regulating and Release Device
P - Dial Thermometer
Q - Tar Baffle
Temp. °F X 100
VOLTS
Power Supply
Water - Inlet
Outlet
Overflow
Compressed Air Supply
Liters

Figure 12. - Modified distillation assay apparatus.

A survey of the yields and properties of products of low-temperature carbonization from representative western coals was made.[69] This study indicates that certain favorable high-volatile coals will yield as much as 40 gallons of low-temperature tar per ton of coal processed.

An improved assay test for determining the yield of products from coal by carbonizing at various temperatures was devised. This test is made in the equipment illustrated in figure 12. The small retort has a capacity of about 6 ounces of coal. It furnishes valuable data on the effect of temperature on products of distillation, for results indicate that the yields shown by the assay can be obtained on a commercial scale by carbonizing the coal in a fluidized state.

Desulfurization During Carbonization with Added Gases

Diverse requirements of coal and its products to meet new and wider applications in industry have focused increased attention towards the development of methods for removing or reducing the undesirable sulfur content of the high-sulfur coals that must be used in the future. Experimental results of an investigation[70] showed that the desulfurizing action, namely, a lowering of the sulfur content in the coke, is increased (1) by addition of ammonia, hydrogen, or nitrogen during carbonization, (2) by introduction of these gases at temperatures lower than the final carbonization or reaction temperature, (3) by continued time of treatment of the coal or coke sample at the reaction temperature with these added gases, and (4) by use of fine-size coke. Carbonization of the coal with added ammonia gas caused a large percentage of the coal sulfur to be converted to volatile sulfur compounds; ammonia was markedly superior to hydrogen, and hydrogen was considerably more effective than nitrogen.

The laboratory investigation suggests that a cheap source of ammonia, such as from ammoniacal liquor, and a simple scrubbing train containing an alkaline solution of suitable composition and concentration might be utilized commercially to effect desulfurization of the solid, liquid, and gaseous products obtained by carbonization, gasification, and combustion of coal.

Ammonium Sulfate Yields

In order to account for differences in commercial yields of ammonium sulfate obtained in different parts of this country, the yields obtained in BM-AGA tests at 800°, 900°, and 1,000° C. from 45 high-volatile A, 14 medium-volatile, and 8 low-volatile coals were averaged by States and compared.[71] Average yields as pounds per ton of coal ranged as follows: High-volatile A coals, 22.5 to 35.9 at 800° C., 19.6 to 30.1 at 900° C., and 15.5 to 27.4 at 1,000° C.; medium-volatile coals, 21.9 to 27.0 at 800° C., 18.0 to 24.4 at 900° C., and 12.0 to 19.2 at 1,000° C., and low-volatile coals, 17.5 to 19.2 at 800° C., 14.0 to 14.9 at 900° C., and 9.0 to 9.2 at 1,000° C. Average yields were: (1) High for Washington, Alabama, and Utah, (2) about the same as the overall average for Kentucky and Oklahoma, and (3) low for Pennsylvania and West Virginia coals.

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[69] Parry, V. F., Goodman, J. B., and Gomez, Manuel, Low-Temperature Distillation Assays of Representative Western United States and Alaska Coals: Colorado School of Mines Quarterly, vol. 45, No. 2A, April 1950, pp. 133-162.

[70] Brewer, R. E., and Ghosh, J. K., Desulfurization of Coal during Carbonization with Added Gases - Quantitative Determination of Sulfur Compounds: Ind. Eng. Chem., vol. 41, September 1949, pp. 2044-2053.

[71] Reynolds, D. A., and Wolfson, D. E., Coal Carbonization: Ammonium Sulfate Yields from Coals of Various Regions of the United States: Bureau of Mines Rept. of Investigations 4526, 1949, 15 pp.

Ammonia yields could not be correlated closely with geological age, moisture content, or nitrogen content, although general trends were noted.

Expanding Properties of Coal During Coking

The sole-heated oven, in which the expansion or contraction of a coal under constant pressure is measured, was used to test 29 coals and 29 blends. The coals tested this year were from the following sources: West Virginia, 9; British Columbia, 9; Kentucky, 4; Pennsylvania, 4; Illinois, 1; Washington, 1; and Alaska, 1.

The coal and blend numbers, dry, mineral-matter-free, fixed carbon content, and expansions expressed on a bulk density of 55.5 pounds per cubic foot and as dry, solid-coal expansion are given in table 9. Expansion on a dry, solid-coal basis is preferred in comparing expansions of two coals or in making correlations of the expanding properties of coal with other coal variables, because it eliminates the differences in expansion caused by varying moisture content and specific gravity of the coal.

Samples of Pittsburgh-bed coal from the Warden mine (p28, q28, and r28) were used for blending with low-volatile coals. The variation in the reported results are supposedly due to the time of storage at the Bureau of Mines before the expansion tests were made. The longer the storage period, the less the contraction. Sample p28 was not tested until 3 months after receipt by the Bureau of Mines.

Pocahontas No. 3, Carswell-mine coal (f75 and g75), is the standard blending coal for use with high-volatile coals. The expansion of Pocahontas No. 3 coal has been observed to increase with storage in sealed containers. Sample f75 was not tested until 5 months after its receipt by the Bureau of Mines. Sample g75 was tested every month, and the increase in its expansion with storage is given in detail in table 10.

TABLE 9. - Expanding properties of coals in the sole-heated oven

Coal No.	Expansion, percent	
	At 55.5 pounds per cubic foot[1]	Dry, solid coal
p28	- 6.6	40.0
q28	-17.4	23.9
r28	-19.2	26.4
f75	+31.9	104.3
g75	+21.5	88.3
f75A	+ 1.0	52.6
f75B	+ 4.3	57.3
a368	- 8.2	39.9
a368A	+ .9	55.2
a368B	+ 3.8	57.7
378	+ 5.3	54.1
378A	- 6.0	41.7
378B	- 3.9	45.3
379	+20.7	78.6
379A	- .3	48.8
379B	+ 4.8	54.4
380[2]	+ 3.7	50.4
380[3]	+ 8.5	59.8
380[2]	+ 3.2	49.0

See footnotes at end of table.

TABLE 9. - Expanding properties of coals in the sole-heated oven - Continued

Coal No.	Expansion, percent	
	At 55.5 pounds per cubic foot[1]	Dry, solid coal
380A[3]	+ 9.4	60.0
380B[3]	+ 8.3	59.3
387[2]	+ .7	43.7
387[3]	+ 6.0	52.6
381	-10.0	39.1
381A	- 4.0	49.5
381B	- 2.6	50.9
382	- 5.6	43.5
382A	- .8	51.2
382B	+ 1.3	55.2
383	- 7.3	41.9
383A	- .8	51.1
383B	+ 1.4	53.9
384	- 6.1	41.3
384A	+ 1.3	53.5
384B	+ 4.3	57.7
386	+39.6	121.7
388	-17.2	31.5
389	-14.3	39.2
392	-19.3	37.4
393	- 8.3	49.5
394	- 7.9	49.3
395	+ 3.7	54.9
398	-13.4	35.8
399	- 5.3	46.1
401	- 6.6	53.9
402	+ 4.6	59.3
413	+11.9	69.6
413A	-16.4	26.4
413B	-13.9	29.2
413C	- 6.0	43.7
415	-19.1	45.1
425	+24.9	89.0
425A	-11.2	38.4
425B	- 3.5	45.4
426	-11.7	33.0
426A	- 1.6	46.8
427	- 7.0	42.8
427A	- .9	53.1
428	- 1.5	47.2
428A	- 8.9	40.9
429	- 4.7	45.9
429A	- 7.2	42.2
429B	-11.0	34.5
429C	- 8.9	41.1

[1] End-of-test contraction for contracting coals; maximum expansion for expanding coals.

[2] Tested as received. Maximum-size particles about 1 inch by 1 inch.

[3] Coal crushed in hammermill.

TABLE 10. - Effect of storage on expansion of Pocahontas No. 3 coal (g75)

Days in storage	Number of tests in average	Average maximum expansion at 55.5 pounds per cubic foot, percent	Increase in expansion from original	Differ significantly; 95-percent range
0	2	21.5	-	Standard
64	2	26.5	5.0	Yes
101	2	24.0	2.5	Yes
128	2	27.6	6.1	Yes
172	2	29.6	8.1	Yes

Statistical analysis shows that differences between average expansions at 55.5 pounds per cubic foot on the same coal will be 1.5 percent or less 95 percent of the time. If this criterion is applied to the differences between averages of two tests on Pocahontas No. 3 coal (g75), the increases in expansion are seen to be significantly different from the standard in each case, indicating that storage of Pocahontas No. 3 coal changes the expanding properties significantly.

A sample of Illinois No. 6 coal (415) was studied in connection with the investigation of the apparent effect of carbon tetrachloride on expansion. This investigation was started because a Pocahontas No. 3 coal, which had been floated on CCl_4 at 1.50 specific gravity, expanded an unusually low amount for its rank. One portion of the Illinois No. 6 coal was cleaned by the heavy media process (magnetite-ore suspension) at 1.55 specific gravity, and another portion was floated on carbon tetrachloride at 1.55 specific gravity. Samples of the raw coal were tested immediately and after storage of several months. Zinc-chloride solution was added to the coal to study the effect of a chloride on expansion. Applying the results of statistical analysis of over 400 test results and based on the 95 percent range, the following conclusions are justified:

1. When the coal is dipped into carbon tetrachloride and thoroughly air-dried before testing, the expanding property is not changed significantly.

2. The presence of carbon tetrachloride as a liquid on the coal when charged lowers the expanding property significantly.

3. Flotation of Illinois No. 6 coal on carbon tetrachloride at 1.55 specific gravity increases the expansion more than does flotation on heavy media of 1.55 specific gravity.

4. The presence of zinc chloride (one pound to approximately 50 pounds of coal) does not lower the expanding property significantly.

Six blends of coke breeze and pitch, four blends of coke breeze and coal, and four blends of coke breeze, pitch, and coal were carbonized in the sole-heated oven, and tumbler tests on the resulting product were made in cooperation with a company interested in agglomerating their coke breeze with pitch or a mixture of pitch and coal. The agglomerated coke breeze could then be used in their carbide furnaces.

The higher tumbler indexes of the agglomerated coke breeze approach those of the lower range of coking-coal blends, but do not compare favorably with the tumbler indexes of the original beehive coke from which the agglomerates were made.

Plasticity of Coals

Plastic properties of 45 coals and 33 blends were determined during the fiscal year. All samples were tested by either the Gieseler and/or Davis plastometer methods; two or more tests on the same sample were made by each test method. A total of 318 tests - 168 by the Gieseler and 150 by the Davis method - was made.

The plastic properties of coals and blends may be conveniently discussed according to characteristic temperature indications of certain stages in the fusion range, the degree of maximum fluidity (dial divisions per minute) developed in the Gieseler test method, and the maximum resistance (pound-inches) observed in the Davis test method. In general, as the rank of the coal increases from high-volatile A to low-volatile bituminous, the fusion range is displaced to higher temperatures, the maximum fluidity becomes less, and the maximum resistance becomes greater. These changes in plastic properties usually correlate well, with corresponding increases in strengths of the cokes produced at 800° and 900° C. from coals of the respective ranks. Variations in chemical and petrographic compositions among individual coals of a given rank have a greater influence on the maximum fluidity and maximum-resistance values than on the temperature range of fusion.

The three coals, a368, 380, and 387, and six blends, f75A, f75B, a368A, a368B, 380A, and 380B, all show good fusion by both test methods and should produce strong cokes. The two samples, 368x and 368y, prepared for testing from Pittsburgh coal (368) by partial carbonization, show complete loss of fusion properties, that is, no resistance in the Davis plastometer test. Samples 368x and 368y should be suitable and samples a368, b368, and 397 unsuitable for gasification by the Lurgi process.

The effect of certain materials on coal-plasticity indexes was studied. Blend 429B, containing 30 percent Sewell coal 429 and 70 percent Pittsburgh coal 28, shows a maximum fluidity of 9,280 dial divisions per minute. As expected, the addition of 11 percent tar increases the maximum fluidity to 12,000, and the addition of 9 percent carbon tetrachloride reduces it to 2,500 dial divisions per minute. This large amount of carbon tetrachloride can conceivably extract some material from the coal or otherwise change its properties in such manner that its plastic properties also are changed.

Coke Properties

Properties and typical analyses of various types of coke were summarized in a recent publication.72/

Oxidizing Properties of Bituminous Coals

The oxidizing properties of 13 coal samples were determined during the year. Twelve samples, after first drying in nitrogen gas at 100° C. in a closed rotary-drum unit, were tested for their spontaneous heating tendencies in oxygen gas in an adiabatic calorimeter. The characteristic rate of oxidation in air at 100° C. of one coal (406) was determined in a steam-jacketed, rotary-drum unit.

Table 11 summarizes the self-heating rates at 70° and 100° C. of the 12 samples as determined in the adiabatic calorimeter and the ratios of these rates to those of

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72/ Brewer, R. E., Solid Fuels 11. Coke: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-37 to 2-39, John Wiley & Sons, Inc., New York, N. Y.

fresh Pittsburgh bed, Warden mine, coal m28, previously tested by the same procedure. Excepting coals 430, 430s, 431, and 431s, which were tested in the sizes given in table 6, all samples were first stage-crushed to 0- to 1/8-inch size. The first five samples listed in the table were tested to determine the effect of prolonged storage on their rates of oxidation. Continued storage of these coals shows that slow oxidation has occurred, as is evidenced by the drop in their self-heating rates. The third test on coal 346 using dry oxygen gas was made to determine the effect of first drying the oxygen completely before admitting it to the coal charge in the calorimeter. Lower self-heating rates are obtained with the dry oxygen. The normal test procedure is to measure the oxygen at room temperature with a wet-test meter before passage to the calorimeter, so that some moisture is always retained in the oxygen at temperatures below 100° C. Pittsburgh coal p28, if fresh, should give the same self-heating rates as the Pittsburgh coal m28 used as the standard of comparison in the table. As would be expected from its rank and long period of storage, the low-volatile Davy Sewell coal 345 oxidizes very slowly. The low-ranking, high-volatile A fresh coal 398 oxidizes very rapidly, as compared with the standard high-volatile A coal m28 which is quite resistant to oxidation. The high-volatile B coal 414, both before and after the drier, shows very rapid rates of self-heating. The effect of finer-size coal in increasing the rate of self-heating is shown by comparing coal 430s with coal 430 and coal 431s with coal 431. The very low rates of self-heating of coals 431 and 431s are due to their high ash content - 63.1 and 63.0 percents, respectively, on the dry-coal basis - as compared with coals 430 and 430s, which contain only 11.5 and 14.1 percent of ash, respectively, on this basis.

TABLE 11. - Self-heating tendencies of coals to heat spontaneously

Coal No.	Condition of coal[1]	Self-heating rate at - 70° C., °C./Hr.	Self-heating rate at - 100° C., °C./Hr.	Ratio of self-heating rate to fresh Pittsburgh bed, Warden mine, coal at - 70° C.	Ratio of self-heating rate to fresh Pittsburgh bed, Warden mine, coal at - 100° C.
346	In storage 13 months	0.50	2.67	0.72	0.97
346	In storage 19 months	.46	2.24	.67	.81
346	In storage 19 months (tested with dry oxygen)	.29	1.10	.42	.40
p28	In storage 6 months	.46	2.70	.67	.98
345	In storage 19 months	.106	1.41	.154	.51
398	Fresh coal	2.79	13.32	4.04	4.83
414	Raw wet feed to driers	5.20	26.0	7.53	9.42
414	Flash dried, 5 minutes at 550° F.	6.00	25.2	8.70	9.13
430	Feed to washery, 0- to 3/8-inch size	1.30	4.86	1.88	1.76
430s	0- to 1/8-inch portion of coal 430	1.58	6.21	2.29	2.25
431	Free discharge refuse, 0- to 3/8-inch	.23	2.70	.33	.98
431s	0- to 1/8-inch portion of coal 431	.29	2.56	.42	.93

[1] The coal sample in all instances was first dried in nitrogen at 100° C. in a closed rotary-drum unit before charging in the adiabatic calorimeter.

The physical properties of 900° C. cokes made by the Bureau of Mines-American Gas Association method from 12 high-volatile A Appalachian coals and their blends with 20 and 30 percent Pocahontas No. 3 coal were studied.[73] The following conclusions were drawn from a study of these data: (1) Increases in the coking power of high-volatile A coals effected by blending with 20 percent Pocahontas No. 3 coal generally were greatest for coals that yielded the weakest coke when carbonized singly; (2) the 1-1/2-inch shatter, 1-inch tumbler, and 1/4-inch tumbler indexes of the cokes from blends of a significant number of the high-volatile coals increased only slightly on raising the proportion of Pocahontas No. 3 from 20 to 30 percent; (3) the 1-1/2-inch shatter indexes of cokes from the high-volatile coals and their blends could be correlated approximately with the 1-inch tumbler indexes, but neither of these indexes was related to the 1/4-inch tumbler index.

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[73] Reynolds, D. A., Coal Carbonization: Effects of Blending Pocahontas No. 3 Coal with 12 High-Volatile A Coals: Bureau of Mines Rept. of Investigations 4552, 1949, 8 pp.; Proc. Am. Gas Assoc., 1949, pp. 725-729.

GASIFICATION OF COAL, COKE, AND LIGNITE

Two chapters covering gaseous fuels and gas producers were prepared for the twelfth edition of Kent's Mechanical Engineers' Handbook. These chapters include data on the physical and chemical properties of gaseous fuels and provide brief descriptions and operating results of typical gas-manufacturing equipment.[74][75]

Lignite

During the year the Lignite Pilot Plant at Grand Forks, N. Dak., continued the investigation of the continuous gasification of lignite in an externally heated annular retort. Two test runs were made with the 310 alloy retort tube installed the previous year and tested in only one run that year. In test No. 12, approximately 85 tons of dried lignite as gasified to give nearly 5.8 million cubic feet of gas in 438 hours of operation. Test No. 13 had a duration of 730 hours during which time over 164 tons of raw lignite as treated to give nearly 7.0 million cubic feet of gas. Though these tests represent an increase of 38 percent in operating time over the previous year and an increase of 61 percent in gas made, there was an increase of only 31 percent in the lignite used. The 310 alloy retort tube appeared to be in entirely satisfactory condition at the end of these test operations.

The marked increase in gas production, with only a moderate increase in hours operated and lignite consumed, is the result of the use of dried lignite in run 12. The use of lignite for gasification has the following advantages over natural lignite: Higher capacity in terms of cubic feet of gas per day and, therefore, reduction in fixed charges per cubic foot of gas made; lower ratio of H_2/CO and, therefore, lower operating costs; more uniform temperature throughout the annular space, and, therefore, more uniform operation; and, more efficient operation in general.

Underground Gasification

The underground gasification of coal has long been advocated as an attractive means of bringing the energy stored in underground coal beds to the surface. With increasing interest in synthetic liquid fuels, the possibility of providing synthesis gas for the various processes by this means has received serious consideration. The advantages which may be realized through underground gasification are: Utilization of dirty coal beds, or coal remaining in worked-out mines; production of energy in a convenient form; reduction of capital expenditure necessary in the mining, transportation, and processing of coal in solid form; and avoidance of the unattractive labor of mining coal.

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[74] Newman, L. L., Gaseous Fuels: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-61 to 2-87, John Wiley & Sons, Inc., New York, N. Y.

[75] Newman, L. L., Gas Producers: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-87 to 2-93, John Wiley & Sons, Inc., New York, N. Y.

An underground gasification plant consists of: A means of inlet to and discharge from the underground system; initial underground development to provide passageways through the coal bed; a source of gasification media, such as compressed air, oxygen, or steam; and means for handling and processing the product gas.

Before a commercial plant may be designed, the factors controlling the process must be determined. The length, width, and shape of the underground gasification unit control the location of the inlet and discharge passages. The optimum rate of gasification in the unit determines the volume of blast medium required and the facilities for handling blast and product gases. Operating characteristics, such as pressure drop through the system, heat loss, and gasification efficiency, must be known. Perhaps the most important factor in the design is the development of techniques for controlling gas quality.

The Bureau of Mines and the Alabama Power Co. have jointly conducted and completed one field-scale experiment in the underground gasification of coal and at present are cooperating in a second field-scale experiment. The Bureau of Mines has, in addition, carried out laboratory-scale investigations relating to the same subject.76/77/

Second Field-Scale Experiment at Gorgas, Ala.

The results of the first field-scale experiment and the laboratory work that followed led to construction of the present larger-scale underground gasification project at Gorgas, Ala. This experiment is being conducted in the Pratt coal bed in an area isolated from the main body of coal by nature and consisting of approximately 100 acres. The coal bed is 42 inches thick and relatively level; it lies under an average cover of 150 feet.

The objectives of this second underground gasification experiment are:

1. To determine the quantity of coal that can be gasified from a given initial combustion zone and the shape and extent of the burned-out areas formed during this gasification.

2. To test the design and installation of types of fixed product-gas outlets, including the seals required.

3. To determine the operational characteristics of the experimental installation under such variation of conditions as the nature of the installation and the progress of the work may indicate to be desirable - for example, the length of passage required, the optimum rate of flow, and the pressure drop.

4. To determine the quality and quantity of the product gas generated and the secondary byproducts evolved under the experimental conditions.

5. To obtain all possible information regarding the action of heat on the overlying strata.

76/ Fies, Milton H., and Elder, J. J., Experiments in the Underground Gasification of Coal: Armed Forces Chem. Jour., vol. III, No. 8, April 1950, pp. 6-9, 30-31.

77/ Synthetic Liquid Fuels. Annual Report of the Secretary of the Interior for 1949. Part I. Oil from Coal: Rept. of Investigations 4651, 1950, pp. 43-49.

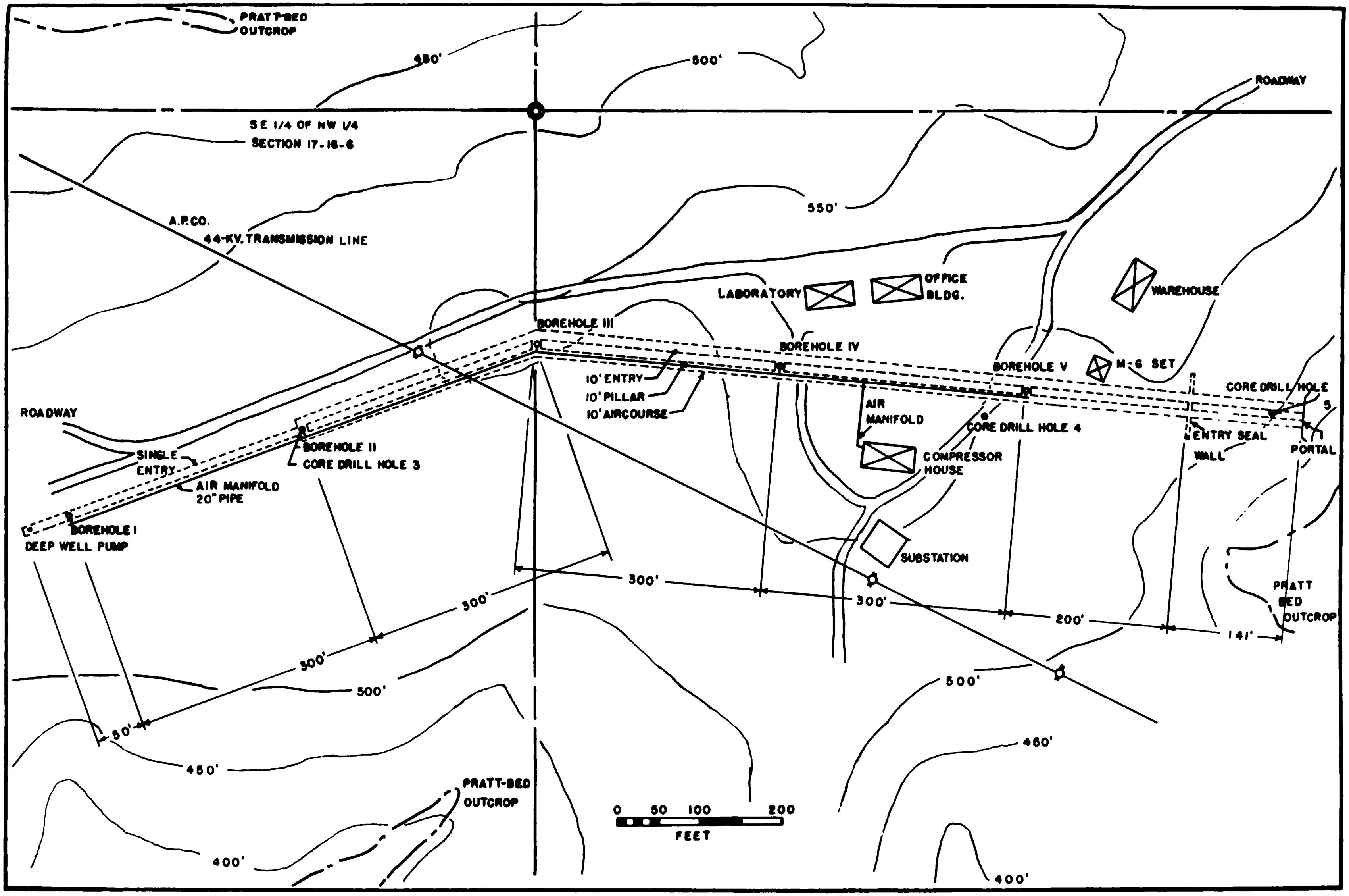

Figure 13. - Site of second experiment in underground gasification of coal, Gorgas, Ala.

6. To develop, without interfering with the foregoing objectives, such fundamental technical and economic information as is likely to be helpful in selecting plant sites, plant installations, and operating processes.

The experimental unit (see fig. 13) consists of a straight-line passage in the coal, with surface connections at one end through an outcrop seal, and at various points along its length through large-diameter vertical boreholes. Small-diameter vertical boreholes were drilled into the coal bed adjacent to the entry, in order to measure the progress of combustion underground. Air is supplied by a reciprocating compressor, rated at 7,200 std. cu. ft. per min. with a discharge pressure of 30 p.s.i.g. Each of the large-diameter boreholes may be used as an inlet or outlet to the underground system.

Combustion was started in the 300-foot length of coal-bed entry, located at the end of the passage farthest from the outcrop. The air blast was maintained for 10 days in one direction, entering at borehole I, and the product gas was discharged at borehole II. During this period the temperature distribution realized and an increased percentage of oxygen in the product gas led to use of periodically reversed blast operation. By this means heat was stored underground and the temperature distribution improved. After several months' operation the oxygen content of the product gas again increased. To remedy this, dry sand was fluidized by means of compressed air and injected into the entry through 6-inch vertical boreholes drilled for that purpose. A total of 156 tons of sand was injected between boreholes I and II, and the operation was materially improved thereby.

In October, November, and the early part of December, the length of time between reversals of flow was increased, and it was found that a combustible gas was being generated on the coal faces and subsequently burned by bypassing oxygen underground. During this period the 20-inch outlet stacks were operated at temperatures of 1,600° to 2,400° F., with gas burning in and above the stack. Coal consumption near the base of the outlet stack increased, and the gaseous products were evolved at temperatures high enough to make possible their use in raising steam or in operating a gas turbine. A large percentage of the heat of combustion of the coal was brought above ground in the effluent gases.

Between boreholes I and II, from March 1949 through March 1950, 5,125 tons of coal were burned. Operating data and the information obtained from the test holes indicate the coal faces receded by 40 feet midway between boreholes I and II and by 65 feet near the boreholes themselves. This means that a total area approximately 130 feet wide at the boreholes, 80 feet wide at the midpoint of the passage, and more than 400 feet long has been burned out, and to date no limit as to the quantity of coal which can be utilized from a given initial passage has been reached. Furthermore, no difficulty has been met in maintaining combustion of the coal underground, and except for minor maintenance shutdowns operation has been continuous on a 3-shift-per-day basis.

From March 1949 to June 1950 the entire operation of the experimental installations was conducted between boreholes I and II. From time to time additional 6-inch churn-drill holes were put down, either along the line of the original entry or in regions where the coal has been burned out. These holes indicated that the overlying strata has been subjected to temperatures high enough to discolor and modify the physical characteristics of the strata, at least, for 15 feet. Where they were drilled along the line of the original entry it was found that roof action in this region had been such that a condition favorable to bypassing the coal faces had been set up. The test holes drilled in regions other than along the original entry and in regions where the coal had been burned out indicated that roof action had been much more favorable to the process.

Experimentation is being carried on to determine the effect of a shorter length of combustion zone, by means of the construction of large boreholes off the line of the original entry at appropriate locations. One such hole was completed and tested and using a combustion face 150 feet in length, a combustible gas was produced for 8 hours. Other experimental work in progress is directed toward the concentration of the combustion zone in a specific location and also to control bypassing of air by plugging underground crevices through the use of fluidized, finely divided solid media. The results so far obtained through this type of experimentation indicate that much better operating conditions can be attained than were realized in the original operation between boreholes I and II.

RESEARCH ON THE PRODUCTION OF SYNTHESIS GAS

Gasification of Pulverized Coal

Pulverized-Fuel-Gasification Pilot Plant

During the year the atmospheric-pressure pilot plant was operated for over 30 test runs.

These runs demonstrated that steam could be preheated to 3,600° F. in the pebble stoves and that, using this steam, the oxygen requirements for gasification of the coal were the lowest for any continuous process reported to date. To secure adequate data for process evaluation, test runs were made using steam at temperatures of 240°, 2,000°, and 3,000° F. As will be noted from table 12, the percent carbon gasified was very good.

A total of 56,000 pounds of coal was gasified at feed rates of 200 to 450 pounds of coal per hour.

TABLE 12. - Averaged requirements per 1,000 std. cu. ft. of ($CO+H_2$) produced

Steam temperature, °F.	Coal, pounds	Oxygen, std. cu. ft.	Steam, pounds	Carbon gasified, percent
2,904	33.3	171	81.1	82.5
1,899	37.3	326	42.5	90.7
238	42.3	394	29.0	83.4

One of the outstanding features of this process is its flexibility as regards the ratio of hydrogen to carbon monoxide, which can be produced by control of the reactant ratios and temperatures. This is shown in figure 14. This feature is of value since it makes the process applicable to a wide range of synthetic operations.

Studies have been made of the variation of the coal-air ratio as the mixture flows through the coal-delivery tube. To measure this ratio, an instrument[78] has been developed which utilizes the change in capacity of a condenser when a mixture of coal and air flows between the plates. The instrument uses two synchronized oscillating circuits, which are coupled inductively. When the two oscillating circuits are in phase, no current flows through the coupling circuit. When the frequency of one circuit is changed by changing the capacity of a condenser in the circuit, current flows in the coupling circuit giving a measure of the change in capacity of the condenser. This current is recorded as a spot on an oscilloscope above a fixed datum, and the spot is moved across the field of the oscilloscope in 1 second.

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[78] Dotson, J. M., Holden, J. H., Seibert, C. B., Simons, H. P., and Schmidt, L. D., New Method Measures the Solid:Gas Ratio in High-Solid Flow: Chem. Eng., vol. 56, October 1949, pp. 128-130.

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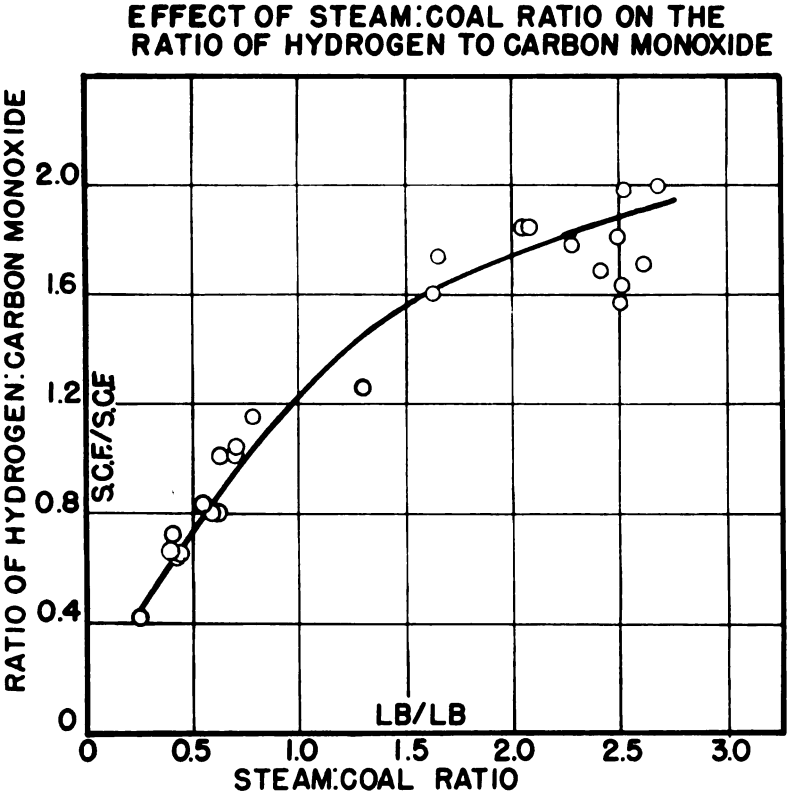

Figure 14. - Effect of steam:coal ratio on the ratio of hydrogen to carbon monoxide.

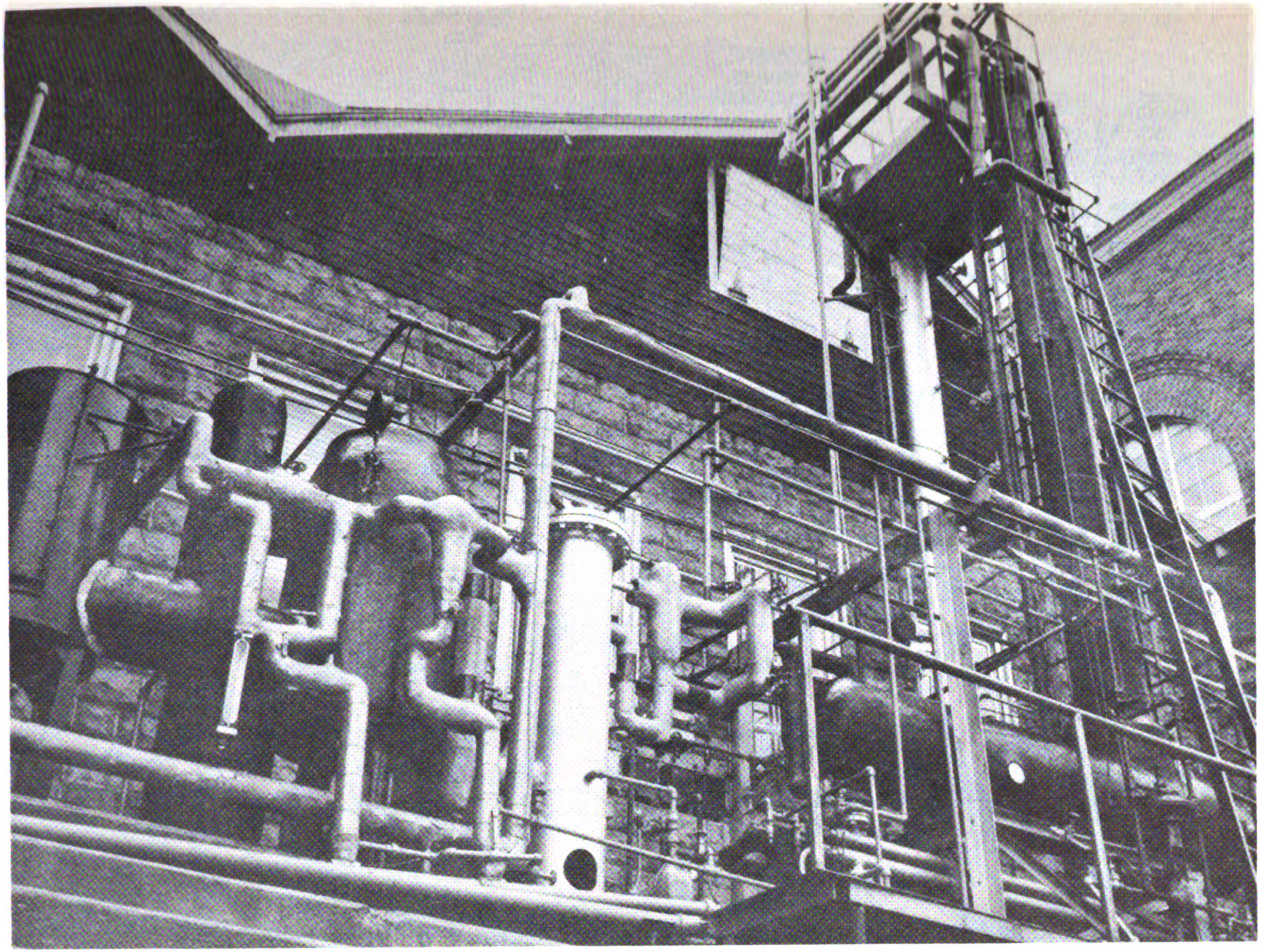

Figure 15. - Gas-purification pilot plant.

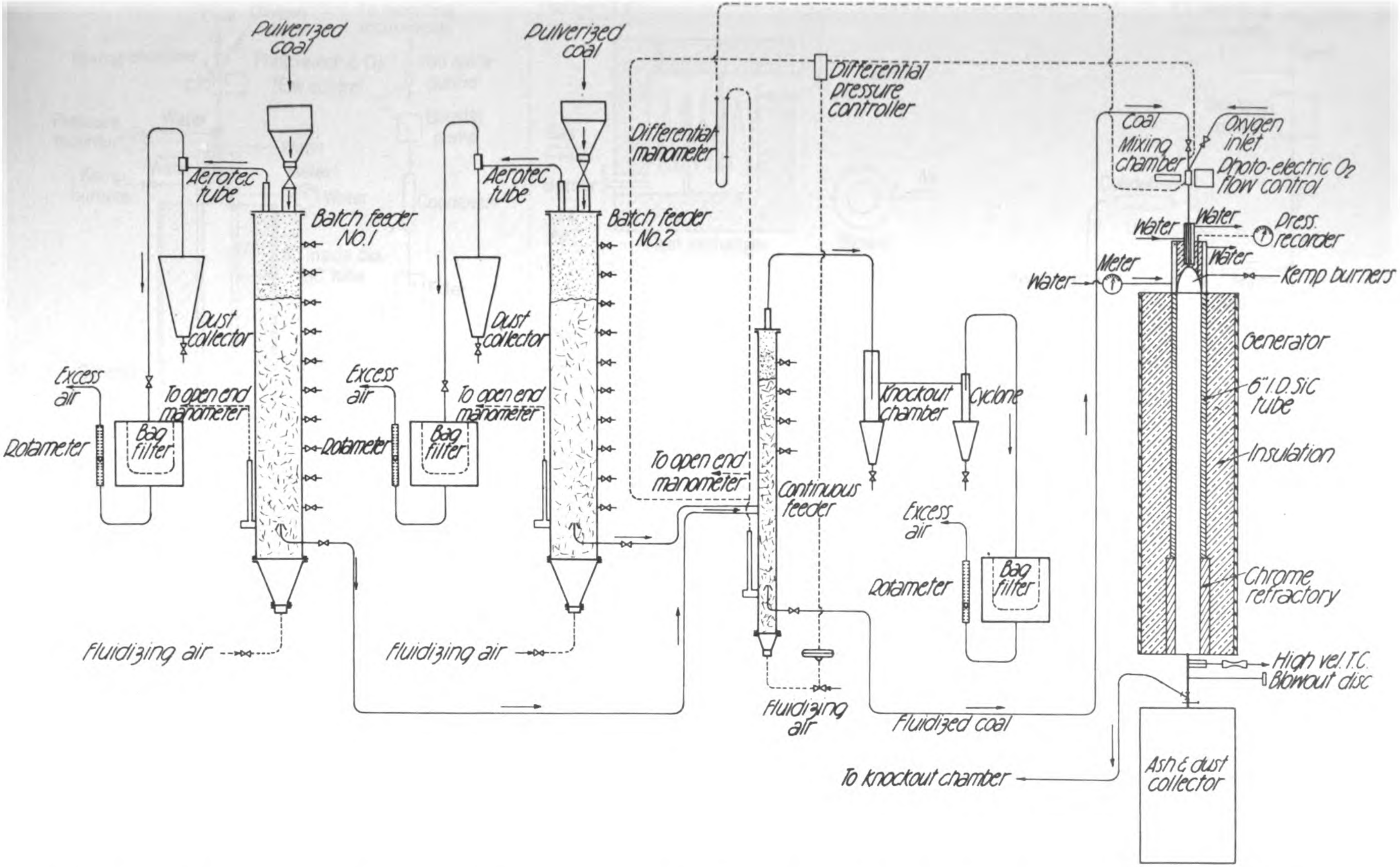

Figure 16. - Flow diagram of pneumatic feeding system and generator for powdered-fuel gasification in laboratory-scale pilot unit.

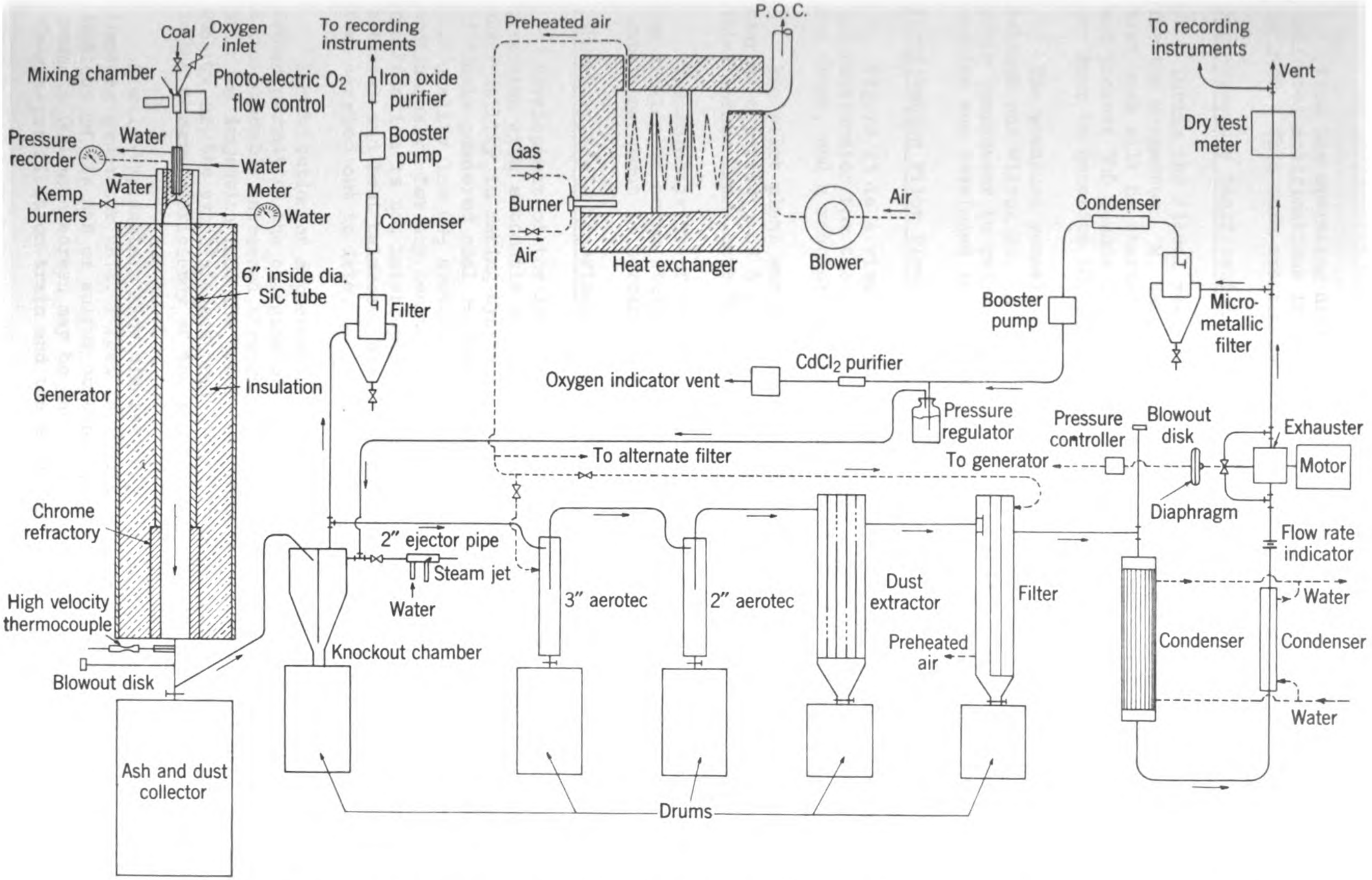

Figure 17. - Flow diagram of generator and synthesis-gas-purification train for powdered-fuel gasification in laboratory-scale pilot unit.

From the operating data obtained from the above described test runs, plans for indicated modifications in the apparatus were drawn up, and rebuilding started early in 1950. This work was completed, and further test work started in June.

High-Pressure Gasification

During the fiscal year 1950, a high-pressure-gasification pilot plant was designed for the Morgantown, W. Va., station. Construction of the unit is well under way, and test work will be started in the fall of 1950. The plant is to operate at 450 p.s.i.g. and process 500 pounds of coal, 4,000 cubic feet of oxygen, and 400 pounds of steam per hour to generate 18,000 cubic feet per hour of carbon monoxide and hydrogen.

The pressure vessels have been furnished under a cooperative agreement with the Babcock and Wilcox Co. A novel feature of the pressurized fluidized feeder is a recycle compressor to recycle gas at 600 p.s.i.g. and 5,000 std. cu. ft. per min. This machine was developed in cooperation with the Sawyer-Bailey Corp.

Purification Pilot Plant

Figure 15 is a view of the gas-purification pilot plant. It includes an absorber and reactivator for the removal of hydrogen sulfide, iron oxide towers, activated carbon drums, and a reactor for the catalytic conversion of organic sulfur compounds.

The pilot plant was originally designed for a gas flow of 500 cubic feet per hour at a pressure of 6 p.s.i.g. The hydrogen sulfide removal equipment and the activated carbon drums have been modified, however, to permit operation at 300 p.s.i.g.

The selectivity of triethanolamine for hydrogen sulfide in the presence of carbon dioxide is being studied at present. The gas used in these runs is inert gas containing about 14 percent carbon dioxide to which hydrogen sulfide is added.

Laboratory-Scale Experimentation

Development of the laboratory-scale down-draft type, atmospheric pilot unit to precision with automatic controls allowed the testing of various types of fuels for their utility in making synthesis gas on commercial scale. Gasifying up to 55 pounds of finely powdered coal in down-draft entrainment in oxygen and superheated steam, a high-quality low-CO_2 synthesis gas up to 1,200 cubic feet per hour can be produced, continuously, for any length of time chosen. The process is easily controlled; the gasification is not interrupted by slagging of the ash, and the results, as judged by material and heat balances, are reproducible. More than 60 gasification runs have been carried out to date.

Substitution of a pneumatic coal-feeding system (fig. 16) for the former Syntron feeder permitted the charging of coal into the generator at a more constant rate and a considerably increased throughput. These, and very thorough mixing with oxygen prior to injection into the generator, resulted in substantial improvements in the quality of the synthesis gas produced, in the percent of coal gasified per pass, and in the thermal efficiency of the process.

A self-explanatory flow diagram (fig. 17) shows the silicon-carbide lined 6-inch-diameter generator unit, 7 feet in length, in which practically any type of coal (regardless of its ash or sulfur content) pulverized so that 70, 80, or 90 percent passes a 200-mesh screen may be gasified. The same flow sheet shows also the synthesis-gas-purification train and the accessory units. Two Kemp low-pressure industrial burners installed in the water-cooled generator head, using premixed natural gas and air, are used to preheat the generator prior to each run to temperatures ranging from 2,400° F. on the top to 1,600° F. at the bottom.

Each run is started by shutting off the preheat burners and charging the powdered coal, carried by the oxygen into the generator head at a velocity of about 120 feet per second, with or without steam in addition to that formed from the moisture in the coal. Gasification of the coal takes place in entrainment (suspension) in a turbulent atmosphere, and the extremely fine carbonaceous residue plus fly ash are carried along by the raw synthesis gas into the subsequent dust-recovery units. Figures 18 and 19 show the external appearance of these units located on two floors below each other and the control board on the upper floor.

In spite of the comparatively small size of the generator, the heat losses were reduced to about 6 percent when coal was charged at a rate of 50 pounds per hour. This has permitted thermodynamic calculation of the operating results, such as composition of the gas made, oxygen requirement per 1,000 cubic feet of synthesis gas, etc., under assumed negligible heat-loss conditions as obtaining in gasification on commercial scale. Calculations have also been made of the percent of carbon in coal gasified and other operating results under thermal-balance conditions, upon the assumption of charging superheated steam at various rates or introducing oxygen at various ratios to coal gasified.

Thus, it was found, for example, that if the 5.7 percent heat loss obtained in a given run were eliminated [Run No. 46: Sewickley coal gasified; coal feed-rate: 44.6 pounds per hour; oxygen to coal ratio: 0.624 and steam to coal ratio: 0.023 pound per pound of dry, ash-free coal. This run was made without steam, except that originating from the moisture in coal. All these are fixed, and the percent of carbon in coal gasified (54.9 percent) was assumed to remain constant.], the percent CO_2 in the gas would drop from 7.1 to 2.7, the volume-ratio of H_2/CO would slightly change from 0.63 to 0.65, the outlet temperature of the make-gas would increase from 1,820° to 2,800° F., the oxygen requirement would decrease slightly from 354 to 325 cubic feet per 1,000 cubic feet of $CO + H_2$, and the yield of synthesis gas would increase from 20.9 to 22.7 cubic feet of $CO + H_2$ per pound of dry, ash-free coal. However, upon increasing the steam to dry, ash-free coal weight-ratio from 0.023 to 1.023 (assuming the addition of 1 pound of steam per pound of dry, ash-free coal, at a temperature of 2,100° F.) under thermal balance conditions, the CO_2 content would go up to 18.0 percent, and the H_2/CO ratio would increase to 1.54. The outlet temperature of the make-gas would drop back to 1,800° F., the oxygen requirement would decrease further to 281 cubic feet per 1,000 cubic feet of $CO + H_2$, the H_2 yield would increase to 26.2 cubic feet per pound of dry, ash-free coal, and 61.7 percent of the carbon in the coal would gasify.

The silicon-carbide lining of the generator was found to have a limited life under the severe conditions to which it is exposed, such as repeated heating and cooling, abrasion, effects of slagging coal ash and strongly oxidizing atmospheres at the top. The upper part of the lining must, therefore, be replaced after a period equivalent to about 100 days of continuous use.

The carbonaceous residue, containing 25 to 30 percent of ash, can be recycled for a second pass through the generator, either alone or in admixture with fresh coal. However, owing to its extremely fine particle size (50 percent less than 7.5 microns) and highly fluffy consistency (bulk density: 2.0 to 2.5 pounds per cubic foot), the residue might find industrial utilization for certain purposes at a lower cost than carbon black. Electron micrographs (fig. 20) revealed a very close similarity to commercial carbon blacks. The carbonaceous residue can also be burned in standard equipment, using powdered coal for underfiring boilers.

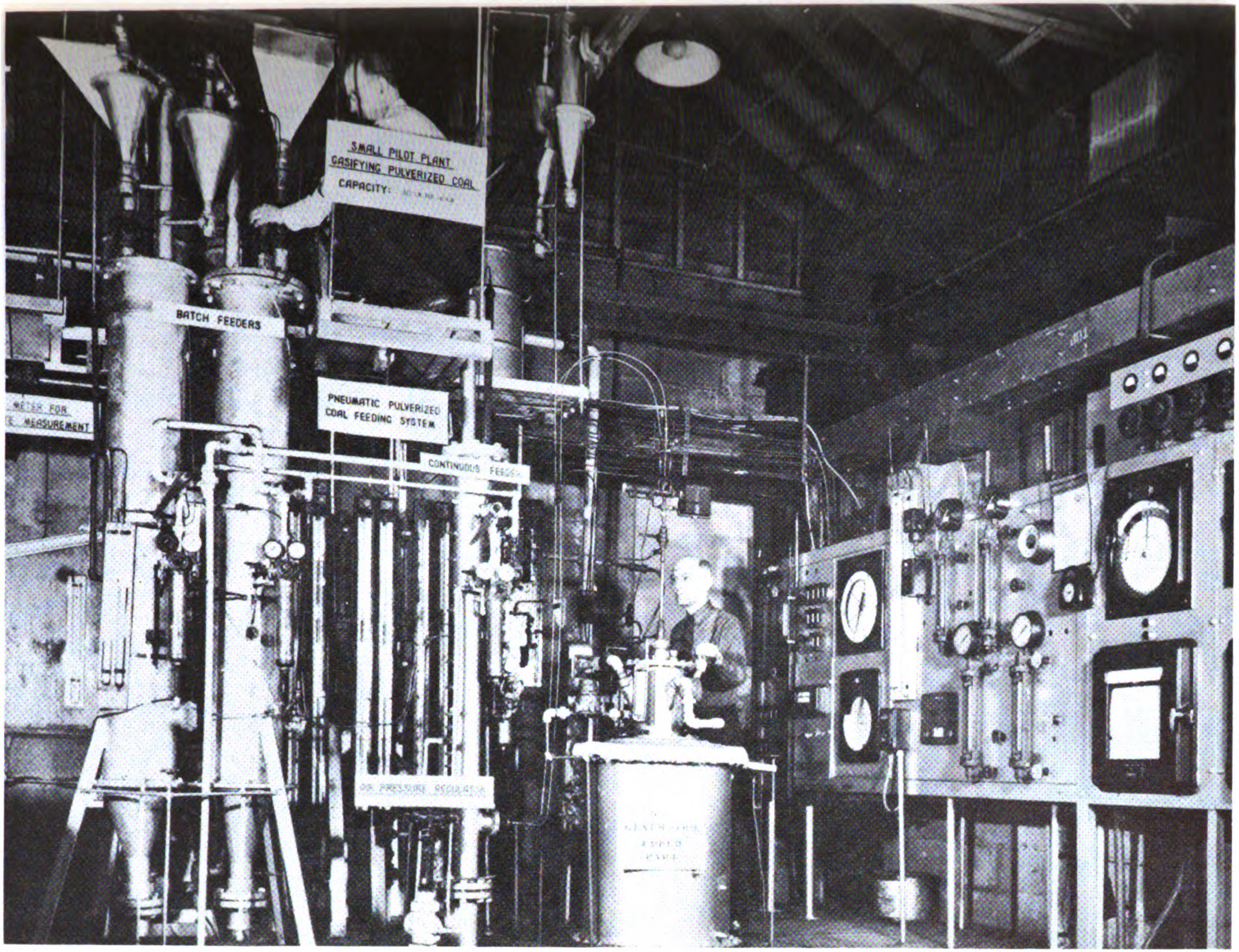

Figure 18. - Laboratory-scale pilot unit (upper floor) for powdered-fuel gasification.

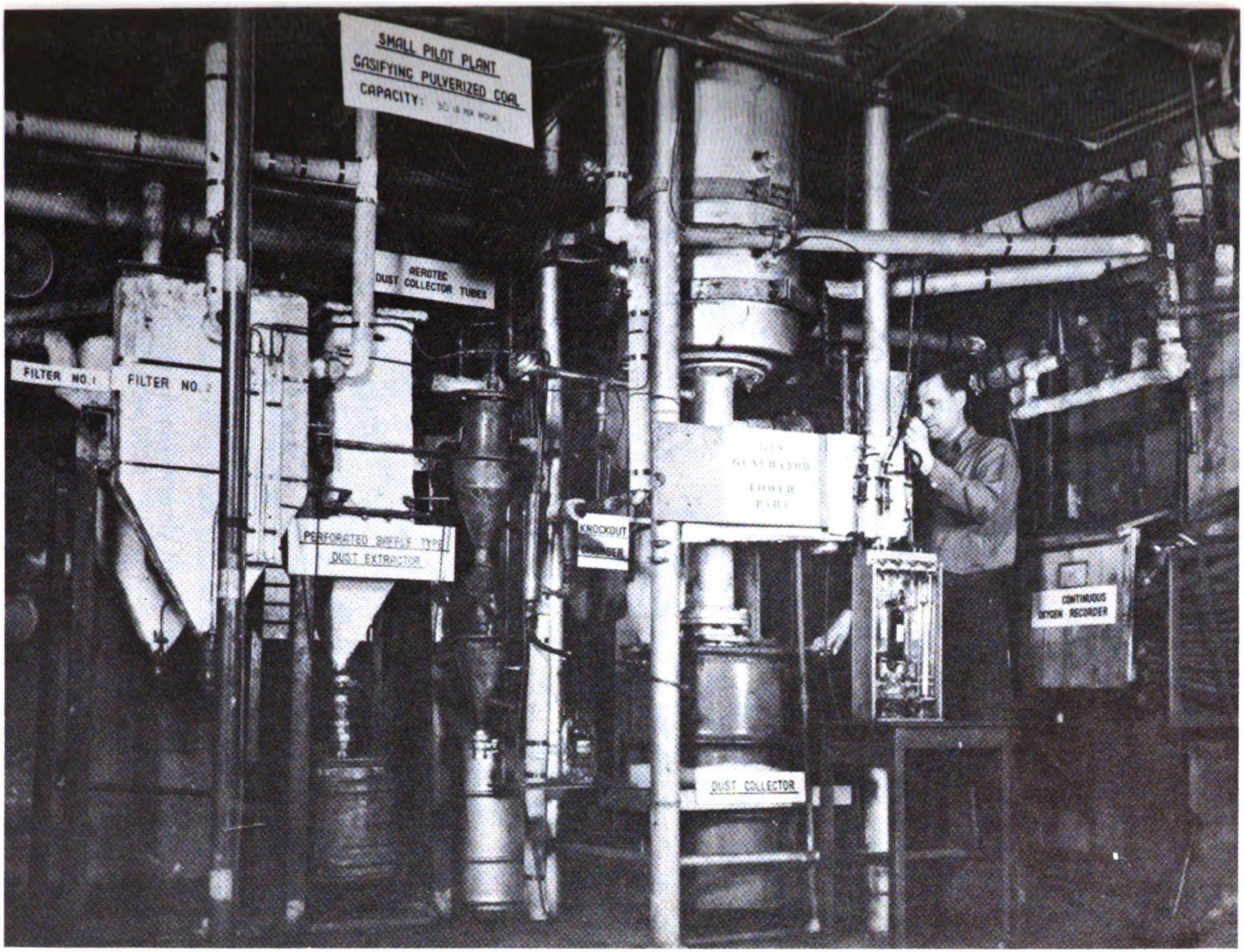
SMALL PILOT PLANT
GASIFYING PULVERIZED COAL
CAPACITY: 30 LB PER HOUR
AEROTEC DUST COLLECTOR TUBES
FILTER NO. 1
FILTER NO. 2
PERFORATED BAFFLE TYPE DUST EXTRACTOR
KNOCKOUT
GAS GENERATOR LOWER PART
DUST COLLECTOR
CONTINUOUS OXYGEN RECORDER

Figure 19. - Laboratory-scale pilot unit (lower floor) for powdered-fuel gasification.

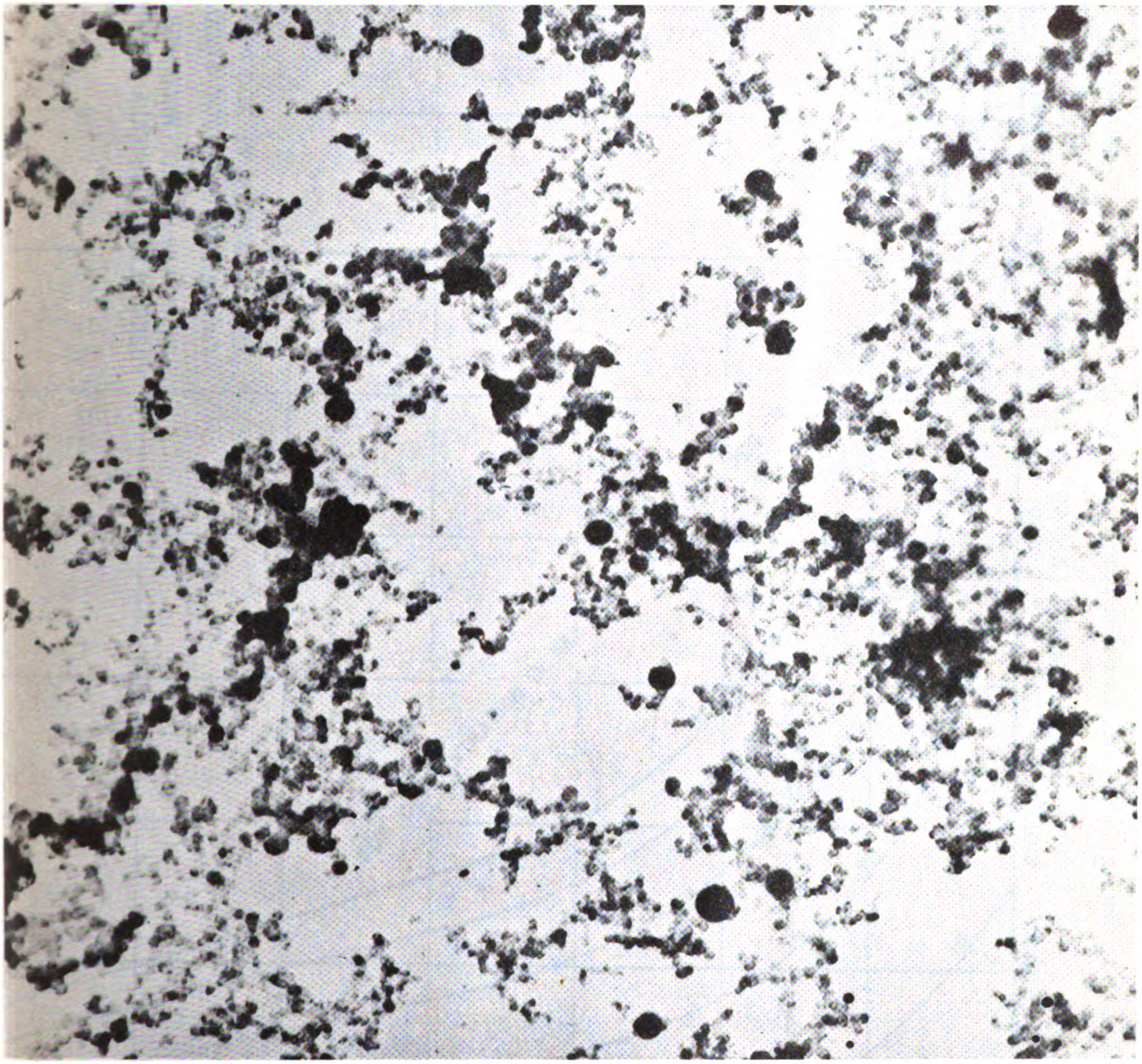

Figure 20. - Electron micrograph of extremely fine carbonaceous residue at high magnification. (X44,000.)

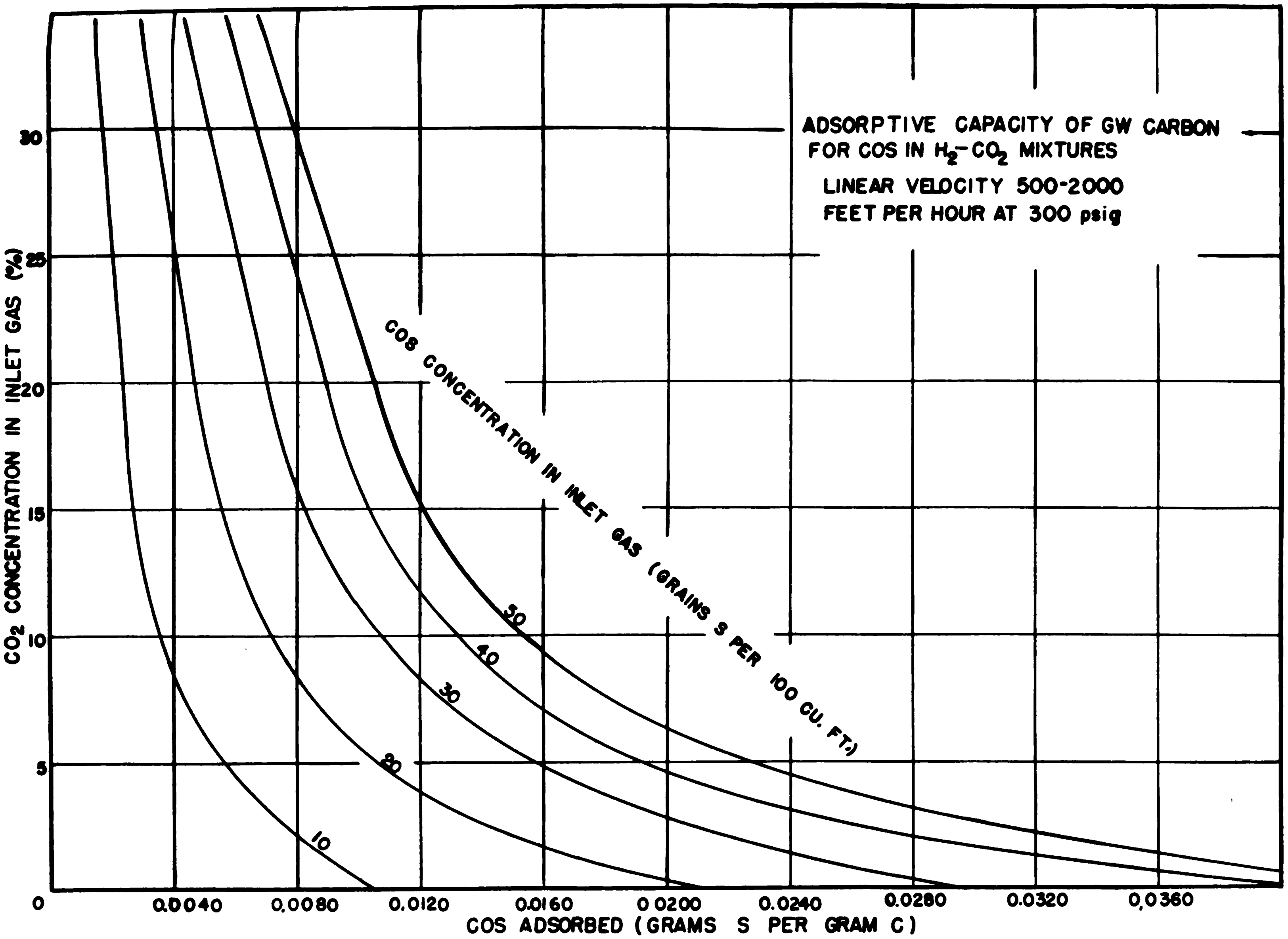

Figure 21. - Adsorptive capacity of GW carbon for COS in H_2-CO_2 mixtures.

Gas Treating and Testing

Sulfur analyses on the gas made in the 50-pound and 500-pound gasifiers indicated that the organic sulfur concentration in the gas is not necessarily proportional to the sulfur content of the coal but is greatly affected by the steam:coal ratio. Other conditions remaining unchanged, increasing the steam:coal ratio greatly decreased the organic sulfur concentration in the gas. Gas made from Sewickley bituminous coal having a sulfur content of 2.4 percent contained approximately 30 grains of sulfur as organic sulfur per 100 cubic feet when steam:coal ratios of about 0.40 were used. Using the same rank of coal having a slightly higher sulfur content of 2.9 percent and steam:coal ratios of 2.0 to 2.7, the organic sulfur content of the make-gas was considerably lower, ranging from 11 to 14 grains of sulfur per 100 cubic feet.

No thiophene sulfur was found in the gas from either gasifier. An experiment in which hydrated lime was added to the coal feed to the small gasifier showed that the addition of 0.038 pound of lime per pound of coal lowered the organic sulfur content of the gas, but had no effect on the hydrogen sulfide. Doubling this ratio of lime to coal further reduced the concentration of organic sulfur and reduced the concentration of hydrogen sulfide in the gas.

Development of analytical methods continued. A turbidimetric method for determining naphthalene in gas using the spectrophotometer and a colorimetric method for nitric oxide were developed. Further investigation of the isatin method for determining thiophene showed that unsaturated hydrocarbons, such as ethylene and propylene, which are known to vitiate the test, do not affect the accuracy of the method if they are present in the gas in amounts less than 3 percent by volume.

An alkalized iron catalyst containing 26 percent sodium carbonate successfully lowered the organic sulfur content of a simulated synthesis gas to less than 0.1 grain of sulfur per 100 cubic feet until 65 percent of the carbonate has been converted to sodium sulfate. Somewhat higher conversion of the carbonate to the sulfate (78 percent) was obtained whenever 100 percent soda ash was used. Experiments with these alkali catalysts were run at 250° to 300° C. and 300 p.s.i.g., employing linear velocities up to 760 feet per hour at 20 atmospheres and space velocities up to 7,000 std. cu. ft. per hr. per cu. ft.

Investigation of a copper-chromium-vanadium catalyst for removing both hydrogen sulfide and organic sulfur from gas was continued. The catalyst, after absorbing about 10 percent of its weight in sulfur, is revivified with air and steam. At a temperature of 450° C. the catalyst reduces the organic sulfur content of the gas to less than 0.01 grain of sulfur per 100 cubic feet while removing 99 percent of the hydrogen sulfide. At a lower temperature of 300° C. the outlet gas contains less than 0.01 grain of sulfur as hydrogen sulfide per 100 cubic feet, but the organic sulfur conversion is somewhat less than that at 450° C., averaging 98.5 percent. Ultrafine purification should be possible by operating two beds in series, the first at a temperature of 450° C. and the second at 300° C.

Numerous tests were made to study the removal of carbon oxysulfide by means of activated carbon at 300 p.s.i.g. Figure 21 graphically presents data obtained from tests using a relatively inexpensive carbon.

The presence of carbon dioxide in the gas has a marked effect on the capacity of the activated carbon for carbon oxysulfide adsorption.

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By combining a specially developed colorimetric method for hydrogen sulfide[79] in gas, with a method whereby organic sulfur compounds in gas are converted to hydrogen sulfide, it is possible to determine the organic content of purified gas in 1 hour.

Previous methods, whereby the gas was burned and sulfur oxides scrubbed from the combustion products, with subsequent gravimetric determination of sulfate, required as much as 48 hours. This reduction in time is important, not only in the laboratory, but especially in commercial plants where very expensive liquid fuels catalysts can be ruined in a few hours if sulfur in the gas becomes excessive.

Vortex Combustion Studies for Powdered-Coal Gasification

As the manufacture of synthetic liquid fuels by the Fischer-Tropsch process requires approximately 30,000 standard cubic feet of hydrogen and carbon monoxide for each barrel of product, any commercial application of this process must be predicated on an abundant source material for the production of synthesis gas. The development of a process which would convert any class of coal into a satisfactory synthesis gas mixture is, therefore, of obvious importance.

One of the most promising methods appears to be the gasification of pulverized coal in suspension in a stream of oxygen and steam, the flow diagram for which is shown in figure 22. Favorable characteristics inherent in such a process appear to be (1) relatively high specific rates of gasification due to the high specific surface of the fuel; (2) the advantages of a continuous process in ease of control and the need of a small plant size for large throughputs; and (3) the use of any class of coal. Suspension gasification of pulverized coal may be classified, in general, in two ways, depending upon the path of the coal particle with respect to the gas: (a) The coal may flow with one or both of the reacting gases; (b) the coal may have relative motion with respect to the reacting gases, thereby making use of the fact that the speed of the diffusion-controlled phases of the gasification reactions is increased. It is this latter type of process which is currently being studied in a reactor of the vortex type in Bureau laboratories.

Preliminary experiments in the vortex reactor (fig. 23) have now been completed. In the absence of methane in the product gas, the chemical reactions in a mixture of carbon, oxygen, and steam at elevated temperatures are:

$$C + O_2 \longrightarrow CO_2 \qquad (1)$$

$$C + H_2O \longrightarrow CO + H_2 \qquad (2)$$

$$C + 2H_2O \longrightarrow CO + 2H_2 \qquad (3)$$

$$C + CO_2 \longrightarrow 2CO \qquad (4)$$

$$CO + H_2O \longrightarrow CO_2 + H_2 \qquad (5)$$

At atmospheric pressure, reaction (1) is assumed to go to completion and the dissociation of steam and carbon dioxide is assumed to be negligible. In the vortex reactor, reaction (4) was found to take place so slowly as not to influence the final product-gas composition. Reaction (5), the water-gas shift reaction, is the only remaining independent equation, since equation (5) equals equation (3) minus equation (2).

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[79] Sands, A. E., Grafius, M. A., Wainwright, H. W., and Wilson, M. W., The Determination of Low Concentrations of Hydrogen Sulfide in Gas by the Methylene Blue Method: Bureau of Mines Rept. of Investigations 4547, 1949, 19 pp.

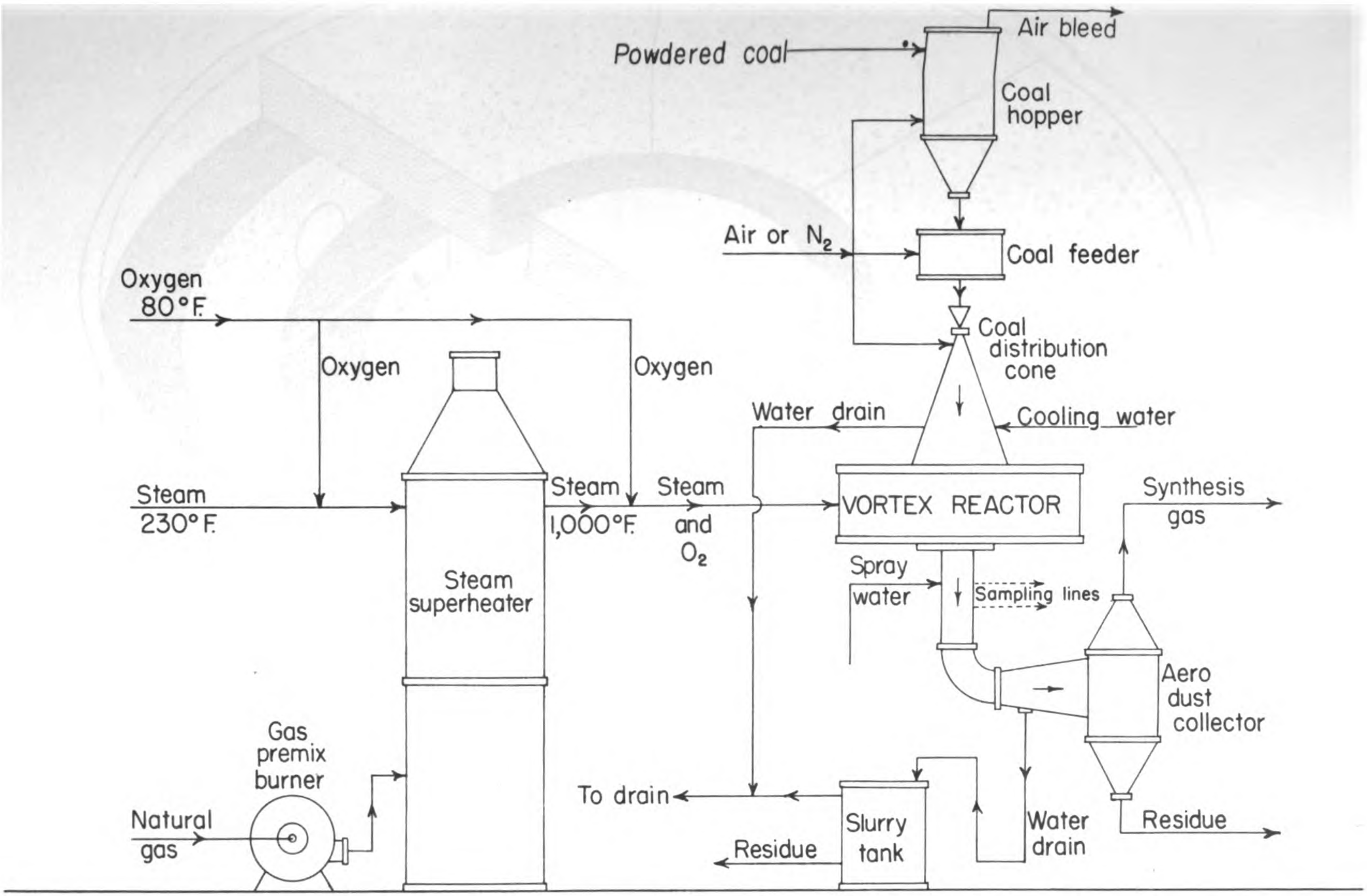

Figure 22. - Flow sheet of vortex gasifier.

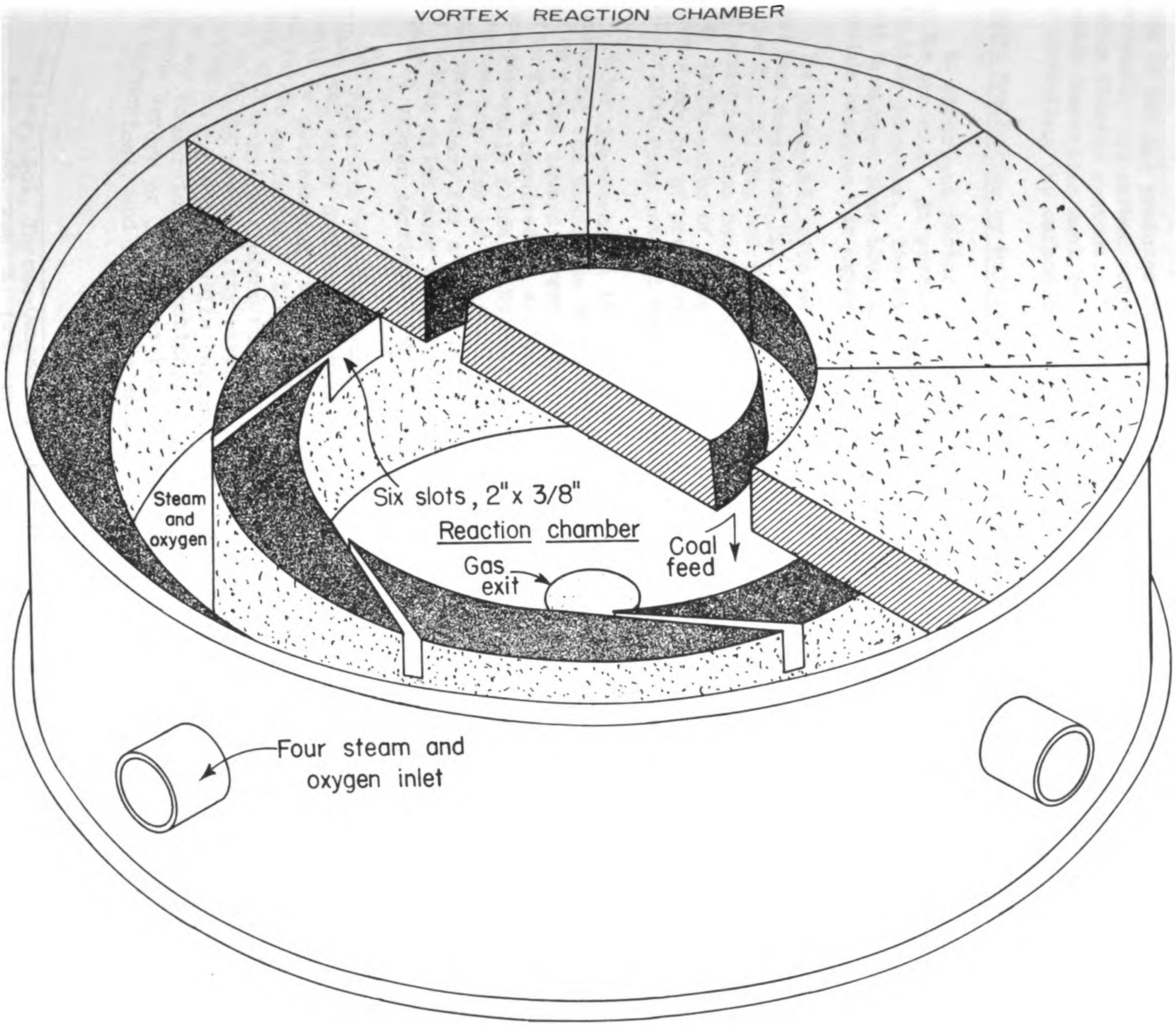

Figure 23. - Vortex reaction chamber.

The percent of carbon dioxide in the dry product gas, the percent steam decomposition, and the ratio of hydrogen to carbon monoxide were calculated as functions of the heat lost by radiation, the preheating of the steam, the oxygen-to-coal ratio, the steam-to-coal ratio, and the percent carbon gasified. These tests confirm the assumption that from the knowledge of the above variables, the approximate composition of the gas produced can be predicted for this reactor. In the preliminary experiments, the carbon conversions were low, and the resulting product gas had a high carbon dioxide content. In order for this reactor to be commercially feasible, the carbon conversion must be increased. For a given set of feed conditions, any means of increasing the carbon conversion will improve the quality of the product gas.

Oxygen Production by Multistage Fractional Permeation

An important factor in the cost of coal gasification is the cost of oxygen used in the process. At present, oxygen is produced commercially almost exclusively by the Linde process. The fact that many organic membranes are appreciably more permeable to oxygen than they are to nitrogen is the basis for experimental work on a possible process for oxygen production.[80]

The permeabilities of a number of thin organic films toward oxygen and nitrogen have been measured.[81] For an ethyl cellulose film, the studies were extended to include CO_2, A, He, and H_2. The enrichment of a binary gas mixture in a single stage of permeation has been calculated for the extreme cases of perfect mixing and no mixing. Application of these results indicates that the use of a fractional permeation process may be of practical importance in effecting the separation of oxygen from air, helium from natural gas, and hydrogen from coke-oven gas, as examples.

Major improvements in the economy of a permeation process could be made by (1) increasing the magnitude of the film permeabilities or (2) increasing the selectivity of the films toward oxygen. Improvements in (1) would permit a decrease in the area of the film required to achieve a given throughput; this might be achieved by finding more permeable films or by making thinner films. Improvements in (2) would not only decrease the size of the plant required but would also decrease the power requirements necessary for a given oxygen production. Such improvements might be achieved by finding films more selective than rubber latex, or by incorporating into films specific oxygen carriers (possibly cobalt chelate complexes).

An apparatus was assembled to investigate the selective permeability of organic films, and eight different commercial materials were studied (table 13). Natural rubber is outstanding for its high permeability and its selectivity; it is, however, subject to oxidation. Ethyl cellulose and polyethylene are promising. Additional work with ethyl cellulose films and with pure gases showed (table 14) that these films had an O_2/N_2 permeability ratio of about 3. Argon is enriched in almost the same proportion as in oxygen, while carbon dioxide becomes enriched to a much greater extent. For any commercial application, it will probably prove desirable to remove carbon dioxide before or just after the first permeation stage. The removal of argon will be impractical, and it will be enriched along with the oxygen.

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[80] Weller, Sol, Thermo-Osmosis of Gases Through a Membrane: Nature (British), vol. 165, Feb. 4, 1950, pp. 199-200.

[81] Weller, S., and Steiner, W. A., Separation of Gases by Fractional Permeation through Membranes: Jour. Appl. Phys., vol. 21, April 1950, pp. 279-283.

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TABLE 13. - Film permeabilities to oxygen and nitrogen at 30° C.

Film	Film thickness, mils	Permeability coefficient[1] Q, cm.^{2}min. $^{-1}$ atm.$^{-1}$ x 10^6		$\frac{Q_{O_2}}{Q_{N_2}}$
		Q_{O_2}	Q_{N_2}	
Ethyl cellulose[2]	0.7	3.25	1.36	2.4
Polyvinyl chloride-acetate	2.0	1.75	1.17	1.5
Vinyl resin + Buna-N rubber	2.0	.70	.58	1.2
Natural rubber latex[3]	7.3	18.1	8.6	2.1
Polyethylene (Plax)	1.5	2.20	.88	2.5
Polyethylene (Bakelite)	.9	2.10	.79	2.7
Polystyrene	1.0	1.31	.44	3.0
Resproid 1404	4.3	3.01	1.08	2.8

[1] Permeability coefficient = (film permeability) (film thickness).

[2] Average of 3 determinations.

[3] Average of 2 determinations.

TABLE 14. - Permeabilities of gases through film EC-12

Gas	Permeability (P),[1] mols x 10^6 cm.$^{-2}$min. $^{-1}$ atm.$^{-1}$	Pgas/P_{N_2}	
		Pure gas studies	Mass spectrometer
N_2	0.075	(1.0)	(1.0)
O_2	.231	3.1	3.2
CO_2	1.07	14.3	[2]32
A	.170	2.3	3.1

[1] Rate of permeation of gas per unit time per unit film area per unit pressure differential.

[2] A blank determination on normal air gave a high CO_2 value.

Synthesis-Gas Production

The synthetic liquid fuels program of the Bureau of Mines at Bruceton, Pa., requires sizeable quantities of hydrogen and mixtures of hydrogen and carbon monoxide. To obtain these gases for operating the pilot plants and experimental units, two plants using Pittsburgh natural gas as a raw material were designed and constructed. One of these plants produces 99-percent pure hydrogen. By the reaction between natural gas and steam over a suitable catalyst, a mixture of hydrogen and carbon oxides is obtained which is passed over a water-gas shift catalyst. The carbon monoxide is thus converted to carbon dioxide, which is removed by monoethanolamine scrubbing. This type of plant has been described in the literature, and a number of installations are in successful operation. The synthesis-gas plant, while using similar equipment, reacts natural gas, steam, and/or carbon dioxide to prepare mixtures of carbon monoxide and hydrogen suitable for use in the Fischer-Tropsch reaction and represents the first plant of this type using industrial-scale equipment to produce synthesis gas of these specifications.

Initial test runs on this plant[82] covered operation at minimum and maximum design rates of $2H_2$:1CO synthesis gas. Studies were also made on production of $1H_2$:1CO synthesis gas. In addition, selected ratios of high carbon monoxide-content gas were produced to determine a range of synthesis-gas compositions available for experimental work. As a result of this work, it has been demonstrated that a wide range of synthesis-gas compositions can be produced on a commercial scale and that the H_2:CO ratio can be controlled as required.

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[82] Clark, E. L., Kallenberger, R. H., Browne, R. Y., and Phillips, J. R., Synthesis Gas Production - Reaction of Light Hydrocarbons, Steam and Carbon Dioxide in Commercial Equipment: Chem. Eng. Progress, vol. 45, November 1949, pp. 651-654.

The Bureau of Mines research program for producing synthesis gas from coal at Morgantown, W. Va., was described.[83]

LIQUEFACTION OF COAL BY DIRECT HYDROGENATION: RESEARCH AND DEVELOPMENT

Function of Solvents and Catalysts in Early Stages of Coal Hydrogenation

Action of Solvents

In most of the early research on the solvent extraction of solid pyrobituminous materials, such as coal, lignite, and peat, mixtures of phenols and tetrahydronaphthalene, in which the ratio of hydrocarbon to phenols in the solvent mixture is customarily about 4:1, were used. In recent work[84] greatly improved yields of soluble materials were obtained by using as the solvent vehicle, dicyclic hydroaromatic compounds containing at least one phenolic hydroxyl group, for example, ortho- and para-cyclohexylphenol and tetrahydronaphthols. About equal parts by weight of solvent and the solid carbonizable fuel being extracted were used. The extraction was carried out using finely ground coal under pressures of 500 to 1,500 pounds per square inch. Temperatures may vary from about 250° C. to as high as 450° C., although it is preferable to operate at approximately 400° C. for typical American bituminous coals. The primary products obtained in this manner were extracts containing little or no ash which were suitable for direct use as fuels, or for use as raw materials for the production of industrial products, such as electrode carbon. Low ash extracts such as these may also be used in hydrogenation processes for the production of synthetic liquid fuels corresponding to gasoline, kerosine, and Diesel fuels from petroleum sources.

In addition to the discovery of these more efficient solvents, the possibility of efficient extraction at atmospheric pressure, with the resultant elimination of expensive, high-pressure equipment, is obviously of practical interest. Phenanthrene was observed earlier to disperse readily about 80 percent of bituminous coal. This fact served as the starting point for an investigation[85] in which determination of the chemical specificity of the solvent and the chemical nature of the extracts was undertaken. The results of these studies showed that in the extraction of bituminous coal, with solvents at their atmospheric-pressure boiling points, carbazole, phenanthridine, 5, 6-benzoquinoline, and certain homologues of these compounds (boiling range, 350° to 360° C.) were equally effective. Anthracene (b.p. 354° C.) and 1, 4-diphenylbutadiene (b.p. 350° C.), however, were shown to be decidedly inferior. Lower-boiling tricyclic compounds, fluorene and diphenylene oxide (b.p., about 300° C.), also proved to be poor solvents, but 1- and 2-naphthol (b.p. 288° and 295° C., respectively) were quite efficient. Hence, it is apparent that the effectiveness of a solvent as an extraction agent for bituminous coal is dependent upon both its molecular structure and its boiling point.

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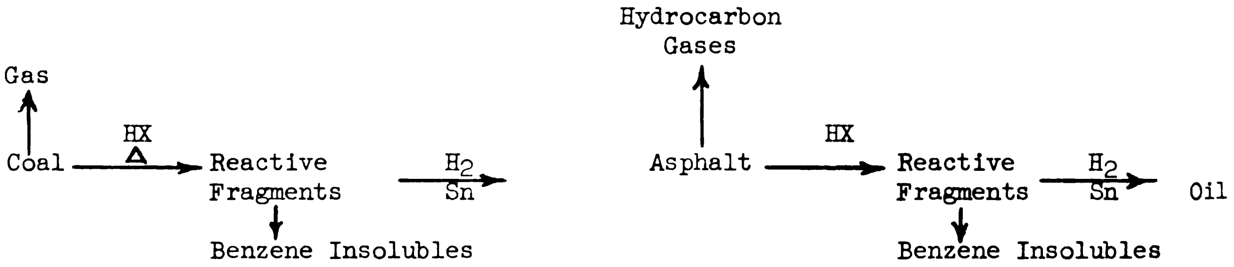
[83] Schmidt, L. D., Cooperative Work of the Federal Bureau of Mines and West Virginia University: The West Virginia Eng., vol. 12, April 1950, pp. 6-8.

[84] Orchin, M., Solvation and Depolymerization of Coal: U. S. Patent 2,476,999, July 26, 1949.

[85] Golumbic, C., Anderson, J. B., Orchin, M., and Storch, H. H., Solvent Extraction of Coal by Aromatic Compounds at Atmospheric Pressure: Bureau of Mines Rept. of Investigations 4662, 1950, 12 pp.

Catalysts for Coal Hydrogenation; Reaction Mechanism

The primary reaction in coal liquefaction by hydrogenation is the production of reactive fragments of the coal structure, which are then stabilized either by polymerization to benzene-insoluble material or by the addition of hydrogen to form benzene-soluble material. It has been assumed that the latter reaction is catalyzed by tin.[86] The resultant benzene-soluble material is largely asphaltic. This asphalt is converted to gaseous hydrocarbons by a noncatalytic reaction and to reactive fragments by a reaction which is catalyzed by halogen-acid-producing substances (HCl, HI, NH_4Cl, etc.). These reactive fragments may (1) recombine to produce asphalt again, (2) polymerize still further to produce benzene-insoluble material, or (3) be stabilized by the addition of hydrogen (catalyzed by tin) to produce an oil. The following diagram indicates this scheme of reactions:



The primary fragmentation of coal, which may be associated with the splitting of carbon-to-oxygen bonds, proceeds with very little gas production. A large fraction of the oxygen in the coal is eliminated, chiefly as water. The conversion of asphaltic material to oil is probably associated with the rupture of carbon-to-carbon bonds.

The use of the proposed mechanism makes possible the interpretation of much experimental data. It can be predicted that: (1) Tin used in the absence of splitting catalyst should behave like no catalyst at all, since the splitting of asphalt to form fragments, which is assumed to precede the hydrogenation stabilization, proceeds very slowly in the absence of halogen-acid-producing substances; (2) hydrochloric acid used alone in an inert atmosphere should cause a large production of benzene-insoluble material, since stabilization of the fragments can then occur only by a disproportionation process; (3) polymerization catalysts, such as phosphoric acid, should produce especially large quantities of benzene insolubles; and (4) any substance which is both a splitting and a hydrogenation catalyst (perhaps hydriodic acid) should result in a high conversion of asphalt and a low production of benzene insolubles. These predictions were verified by experiment.

The above mechanism for the conversion of coal to oil does not explain the unique excellence of tin as a coal-hydrogenation catalyst, under a great variety of conditions of temperature and pressure and irrespective of the chemical form in which tin is used.

Experiments in batch autoclaves have shown, however, that stannous sulfide may be, thermodynamically speaking, the "least common denominator" for all forms of tin under coal-hydrogenating conditions. Calculation of the free energy changes involved shows that stannic oxide can be completely converted to stannous sulfide at 700° K., provided, the hydrogen sulfide pressure is kept above 0.06 atmosphere. This condition is almost certainly met in most coal hydrogenations.

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[86] Weller, Sol, Clark, E. L., and Pelipetz, M. G., (Coal Hydrogenation Catalysts) Mechanism of Coal Hydrogenation: Ind. Eng. Chem., vol. 42, February 1950, pp. 334-336.

The search for a cheaper and more readily available catalyst than tin continued.[87] The only material, that was found to be as effective as tin (when catalysts are added as powdered solids) for the hydrogenation of coal, is germanium, which is even less economically desirable. Table 15 shows that at approximately 2,000 p.s.i.g. reaction pressure zinc bromide and lead bromide were markedly catalytic, resulting in liquefactions of 75 and 78 percent, respectively. It seems probable that the effect is due to the hydrogen bromide liberated under reaction conditions, which may act as a hydrogen donor as hydrogen iodide does. The study of iron catalysts for possible use in the demonstration plant has shown dried "red mud" to be promising at reaction pressures of approximately 7,500 p.s.i.g.

TABLE 15. - Hydrogenation of Bruceton bituminous coal

(In laboratory autoclaves at 450° C., 1 hour, 1,000 pounds per square inch initial H_2 pressure, in absence of vehicle. Pyrex glass liner used to avoid catalysis by metal walls.)

Run No.	Catalysts	Percent of liquefaction	Asphalt per unit of liquefaction
860...	1 percent Sn + HI 0.55 percent NH_4Cl	87.5	0.21
850...	HI 0.55 percent NH_4Cl	66.6	.39
862...	HCl 0.55 percent NH_4Cl	40.2	.48
518...	0.55 percent NH_4Cl	40.8	
1169..	1 percent Mo as MoS_2 + 0.5 percent NH_4Cl	49.0	.39
1115..	1 percent W as WS_2	62.1	.54
1150..	1 percent Ni as Ni-kieselguhr + 0.5 percent NH_4Cl	73.3	.56
964...	1 percent Ge as GeO_2 + 0.5 percent NH_4Cl	86.8	.51
1158..	1 percent Ni as $NiCl_2$	40.0	.28
1159..	1 percent Co as $CoCl_2$	50.6	.53
1173..	1 percent Zn as $ZnCl_2$	44.2	.27
1162..	1 percent Zn as $ZnBr_2$	75.2	.22
1160..	1 percent Pb as $PbCl_2$	49.0	.43
1161..	1 percent Pb as $PbBr_2$	78.5	.45
1175..	1 percent Fe as $FeCl_2$	61.1	.26
845[1].	1 percent dried "red mud"[2]	83.5	.49
1181[1]	1 percent dried "red mud"[3]	79.0	.46
1189[1]	5 percent dried "red mud"[3]	94.2	.41

[1] Rock Springs coal used at 3,700 p.s.i. initial H_2 pressure.

[2] Obtained through the courtesy of the Aluminum Co. of America; this material is the equivalent of Bayermasse, used extensively in German coal-hydrogenation plants.

[3] Type M. obtained from Bureau of Mines, Louisiana, Mo.

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[87] Weller, Sol, Pelipetz, M. G., Friedman, Sam, and Storch, H. H., Coal Hydrogenation Catalysts - Batch Autoclave Tests: Ind. Eng. Chem., vol. 42, February 1950, pp. 330-334.

Bench-Scale Process Development

Dependence of Coal Hydrogenation on Operating Variables

A full understanding of the mechanism by which coal is converted to oil and gas during its high-pressure, high-temperature hydrogenation must rest on a critical interpretation of the manner in which external variables influence the rate and course of the coal-hydrogenation reactions. Recent autoclave experiments[88] that extend the results of previous investigations on the kinetics of coal hydrogenation have resulted in the following observations.

In both the presence and the absence of an asphalt-free heavy-oil vehicle, the effect of hydrogen pressure on dry Bruceton coal at 450° C. for 1 hour (tin catalyst) is most important for initial pressures up to about 2,000 pounds per square inch. (Under reaction conditions, the hydrogen partial pressure is 2 to 2.5 times the initial pressure.) Above 2,000 pounds per square inch, under the reaction conditions used, an increase in initial hydrogen pressure played only a small role in determining the progress of the reaction. The hydrocarbon gas production and the hydrogen consumption are appreciably lower in the absence of vehicle. Part of this behavior is doubtless due to a production of gas by the hydrogenation of the heavy-oil vehicle.

In the range of 400° to 500° C., hydrogenation of dry Bruceton coal for 1 hour at 1,000 to 2,000 pounds per square inch hydrogen pressure (tin catalyst) showed that an increase in reaction temperature resulted in a decrease in asphalt content of the products. Corresponding increases in hydrocarbon gas production and in hydrogen consumption were also observed with increasing temperature. Within experimental error, the composition of the hydrocarbon gases produced was the same and was independent of temperature; this composition corresponded to approximately 30 percent CH_4, 25 percent C_2H_6, 25 percent C_3H_8, 15 percent C_3H_{10}, and 5 percent C_5H_{12}. It is interesting to note that coal can be almost completely liquefied (86 percent) under conditions where the principal hydrogenation product is asphalt, very little gas or oil being formed.

A study of the results obtained from the hydrogenation of dry Bruceton coal at a hydrogen pressure of 2,500 pounds per square inch and 400°, 430°, or 450° C. (tin catalyst) reveals that the liquefaction of coal proceeds rapidly in the absence of vehicle, even at 400° C.; at this temperature about 1 hour is sufficient to produce liquefaction greater than 85 percent. The hydrogenation of asphalt, however, is very slow at 400° C. At 430° C. the asphalt product is hydrogenated at an appreciable rate, and at 450° C. the asphalt hydrogenation proceeds almost as rapidly as does the liquefaction of coal. A variable contact-time unit for the hydrogenation of coal is shown in figure 24.

There has been a good deal of speculation as to the function of a vehicle in the coal-hydrogenation process. One of the most popular theories has been the suggestion that a vehicle, such as tetralin, functions by supplying hydrogen to the coal and that the high-pressure hydrogen present in the system serves to regenerate the tetralin. A study of the results obtained from the above experiments, as well as from those autoclave experiments[89] in which Bruceton coal was hydrogenated in the presence

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[88] Pelipetz, M. G., Kuhn, E. M., Friedman, S., and Storch, H. H., Studies of the Kinetics of Coal Hydrogenation: Bureau of Mines Rept. of Investigations 4546, 1949, 9 pp.

[89] Orchin, Milton, Goldbach, G. L., Wolak, Margaret, and Storch, H. H., Coal Hydrogenation: The Effect of Variations in the Coal-to-Vehicle Ratio: Bureau of Mines Rept. of Investigations 4499, 1949, 10 pp.

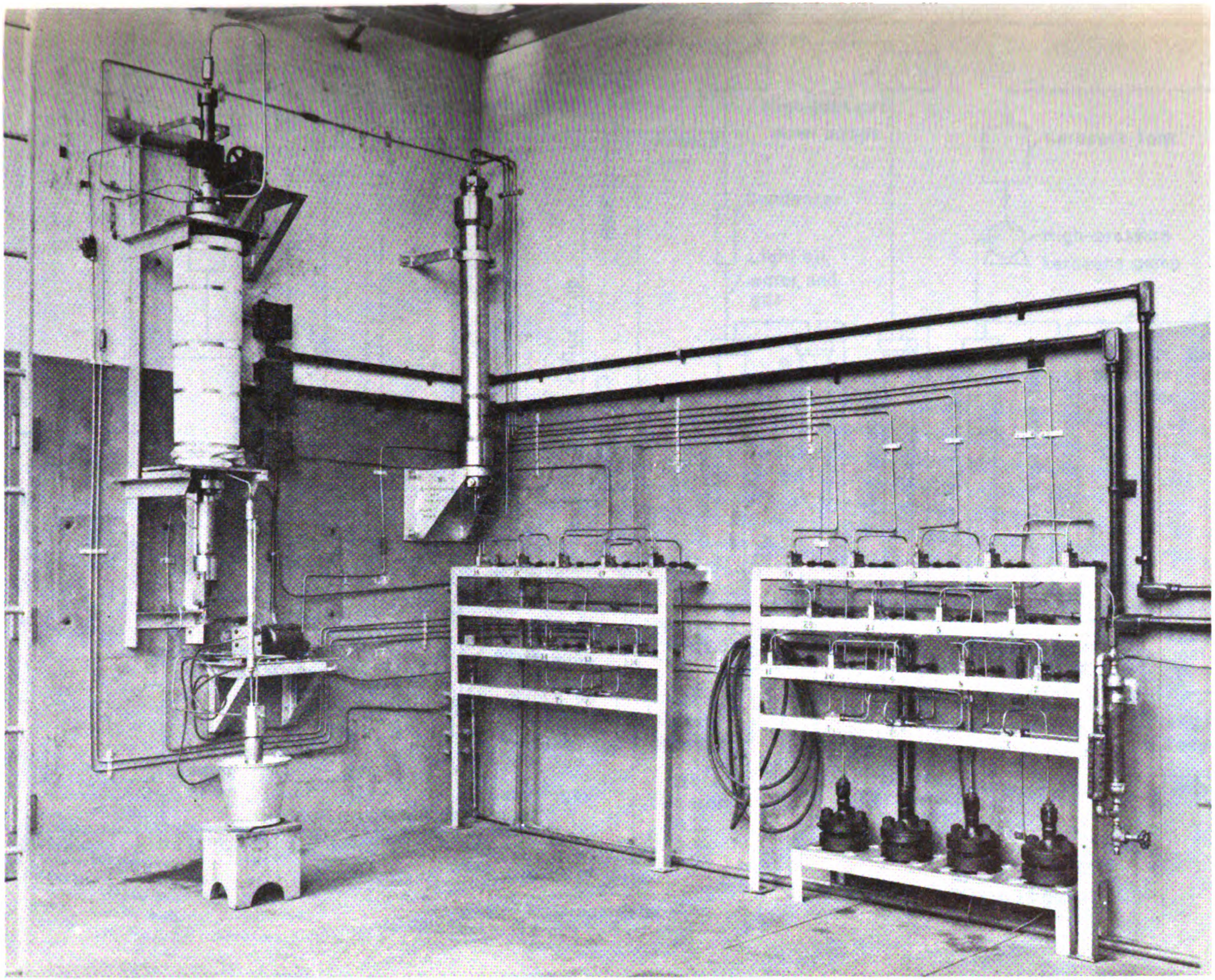

Figure 24. - Variable contact-time unit for hydrogenation of coal.

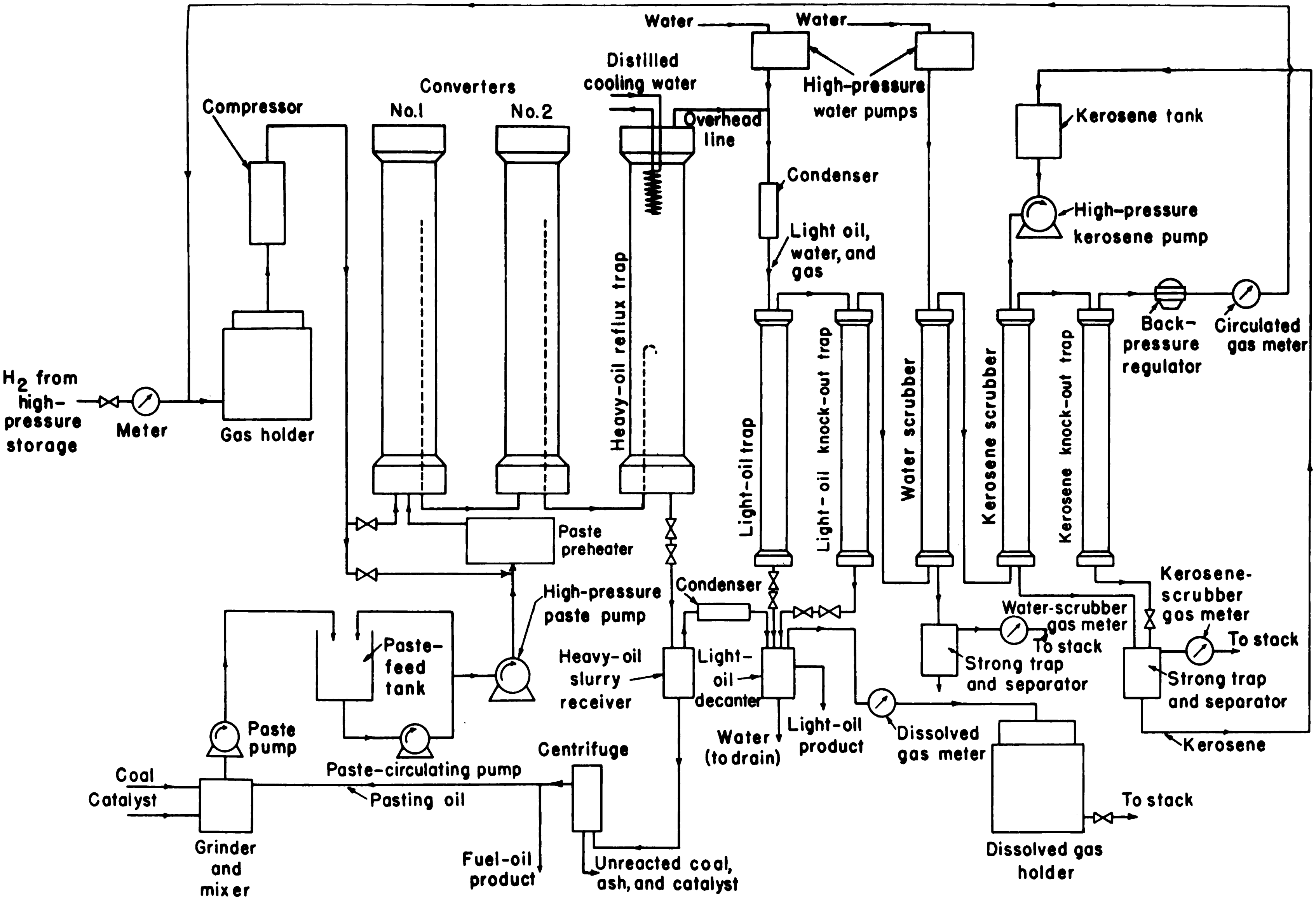

Figure 25. - Schematic flow diagram of coal-oil experimental plant.

of tetralin, largely substantiate the reaction mechanism described earlier. The experiments using tetralin indicate that the extent and rate of liquefaction of coal is not greatly dependent on the vehicle functioning as a hydrogen donor to the coal substance and that the slow step in the process is not likely to be the regeneration of such a vehicle. In the experiments using a heavy-oil vehicle, the progress of the liquefaction is almost unaffected by the presence of the heavy oil, but the rate at which asphalt is converted to lighter products is very much slower in the presence of this vehicle. This behavior will be very important in determining the relative merits of the conventional liquid-phase coal-hydrogenation process and one involving dry coal.

Hydrogenation of Coal in the Absence of Solvents

German practices in the high-pressure hydrogenation of coal using liquid-phase vehicle (Bergius-I.G. process) have been thoroughly studied. Many features of this process indicate that potential advantages may be realized by effecting a reaction between coal and hydrogen in the absence of such solvents. In this manner, the equipment required to condition heavy-oil let-down for use as pasting or injection oil with the coal feed could be eliminated. Reaction space used by the oil portion of the coal-oil feed mixture could be used for the reaction of additional coal, and the removal of unreacted coal as a dry material, instead of a mixture with oil, would materially decrease the capital and maintenance cost of the hydrogenation plant. In addition, work in batch autoclaves has indicated that the heavy oil exercises a deleterious effect on the reaction.

Bench-scale hydrogenation of uncatalyzed Wyoming bituminous coal in an agitated or fluidized bed[90] resulted in an oil yield of 20 to 27 percent and hydrocarbon gas yield of 20 to 27 percent of the moisture- and ash-free coal at hydrogen pressures of 250 to 1,000 pounds per square inch and temperatures of 500° to 600° C. Hydrogenation of coal using a fluidized coal bed thus offers a possible means of dry coal processing at relatively low pressures. Products of the fluidized bed hydrogenation are oil, hydrocarbon gases, and a char residue suitable for use directly as fuel or for gasification to produce synthesis gas.

Pilot-Plant Operations

A 9-month series of tests studying the manufacture of heavy fuel oil by high-pressure hydrogenation of Bruceton and Rock Springs bituminous coals was completed in the experimental liquid-phase pilot plant.[91] While one aim of this series of tests was the production of fuel oil meeting the requirements of the Bunker C or A.S.T.M.-6 grade, a sufficiently wide range of primary variables was investigated so that a comprehensive process variable study could be made. Included were the effects of contact time, reaction temperature, and concentration of the coal-oil feed paste on product viscosity, oxygen content, residual asphalt, sulfur content, and nondistillable soluble oil content for these coals using stannous sulfide and molybdenum trioxide as catalysts. A typical flow sheet of the pilot plant used in these studies is shown in figure 25. Results of the pilot-plant operations yielded, in addition to the above process variable study, tentative relationships between the several primary variables and hydrogen absorption, soluble oil yield, and light hydrocarbon-gas formation, as well as an approximate correlation of these parameters with product viscosity.

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[90] Clark, E. L., Pelipetz, M. G., Storch, H. H., Weller, S., and Schreiber, S., Hydrogenation of Coal in a Fluidized Bed: Ind. Eng. Chem., vol. 42, May 1950, pp. 861-865.

[91] Elliott, M. A., Kandiner, H. J., Dallenberger, R. H., Hiteshue, R. W., and Storch, H. H., Hydrogenation of Bituminous Coal in Experimental Flow Plant - Process Variable Study: Ind. Eng. Chem., vol. 42, January 1950, pp. 83-91.

Equilibria involving a single equation relating the product and reactant concentrations are readily treated by using the "degree of reaction" as a parameter. Where two or three equations are necessary to describe the concentration relationships in a system, this procedure may still be used, but the development and solution of the algebraic equations encountered are considerably more difficult. In really complex systems, such as that encountered in a continuous coal-hydrogenation process, the initial problem of selecting a consistent set of suitable chemical equilibria becomes very involved, and the labor of mathematical solution is almost prohibitive. The development of a simple method to accomplish this purpose92/ has greatly facilitated the analysis of complex equilibria existing among any number of chemical species in a single (gas) phase with or without the presence of a single solid phase.

Separation and Identification of Products of Coal Hydrogenation

Phenols in Coal-Hydrogenation Oils

Phenolic compounds of a wide variety are potentially available in huge amounts from coal-hydrogenation product oils.93/ It is, therefore, of considerable interest to determine the composition of the complex tar-acid fractions in which these phenols are found. Such mixtures are composed largely of homologous and isomeric phenols, and separation by the usual procedures of precise fractional distillation and fractional crystallization is difficult.

Nevertheless, these techniques, plus the invaluable assistance of qualitative and quantitative analyses94/ by means of the infrared spectrometer, have led to the characterization of those product oils distilling to about 200° C. Analysis of the higher-boiling fractions, however, is considerably more difficult because of the greater complexity of the tar-acid mixtures and because no infrared reference spectra are available. To gain an insight into the character of these very complex mixtures of acidic, aromatic hydroxy compounds, the countercurrent distribution technique, which was developed in other laboratories for the separation and analysis of small quantities of closely related compounds (such as antimalarial drugs and penicillin preparations), is being applied.

In the countercurrent distribution procedure, a mixture of phenols was distributed between two immiscible solvents in successive stages until separation into its components was achieved. During this investigation, it was observed that a relationship existed between partition coefficients and ionization constants of phenols and between these constants and the chemical configuration of phenol homologues.95/ In this way, it was possible to isolate individual or groups of closely related tar acids; 4-indanol, 5-indanol, and p-phenylphenol were isolated and identified as were 3-methyl-5-ethyl-phenol and o-phenylphenol. Thus, such a technique, when applied to a synthetic mixture of 3,5-xylenol, 2,5-xylenol, and 2,4-xylenol or a mixture of

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92/ Kandiner, H. J., and Brinkley, S. R., Jr., Calculation of Complex Equilibrium Relations: Ind. Eng. Chem., vol. 42, May 1950, pp. 850-855.

93/ Woolfolk, E. O., Orchin, M., and Dull, M. F., Phenols in Oil Obtained from Hydrogenation of Coal - Separation and Identification: Ind. Eng. Chem., vol. 42, March 1950, pp. 552-556.

94/ Friedel, R. A., Pierce, L., and McGovern, J. J., Infrared Analysis of Phenol, Cresols, Xylenols, and Ethylphenols: Anal. Chem., vol. 22, March 1950, pp. 418-420.

95/ Golumbic, C., Orchin, M., and Weller, S., Partition Studies on Phenols. I. Relation Between Partition Coefficient and Ionization Constant: Jour. Am. Chem. Soc., vol. 71, August 1949, pp. 2624-2627.

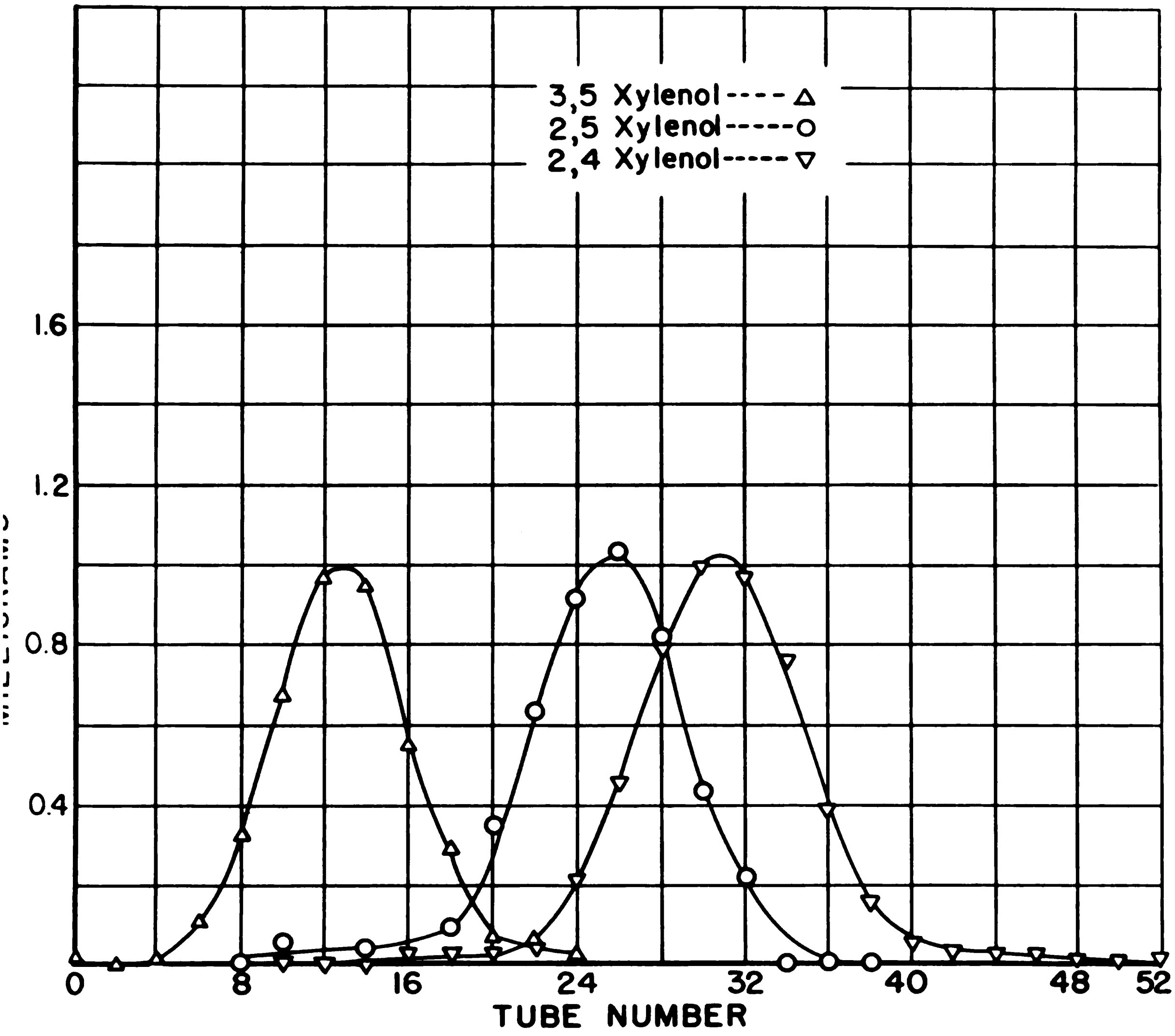

Figure 26. - Separation of xylenols: System, cyclohexane/phosphate buffer pH 11.08; sample, 10 mg. each.

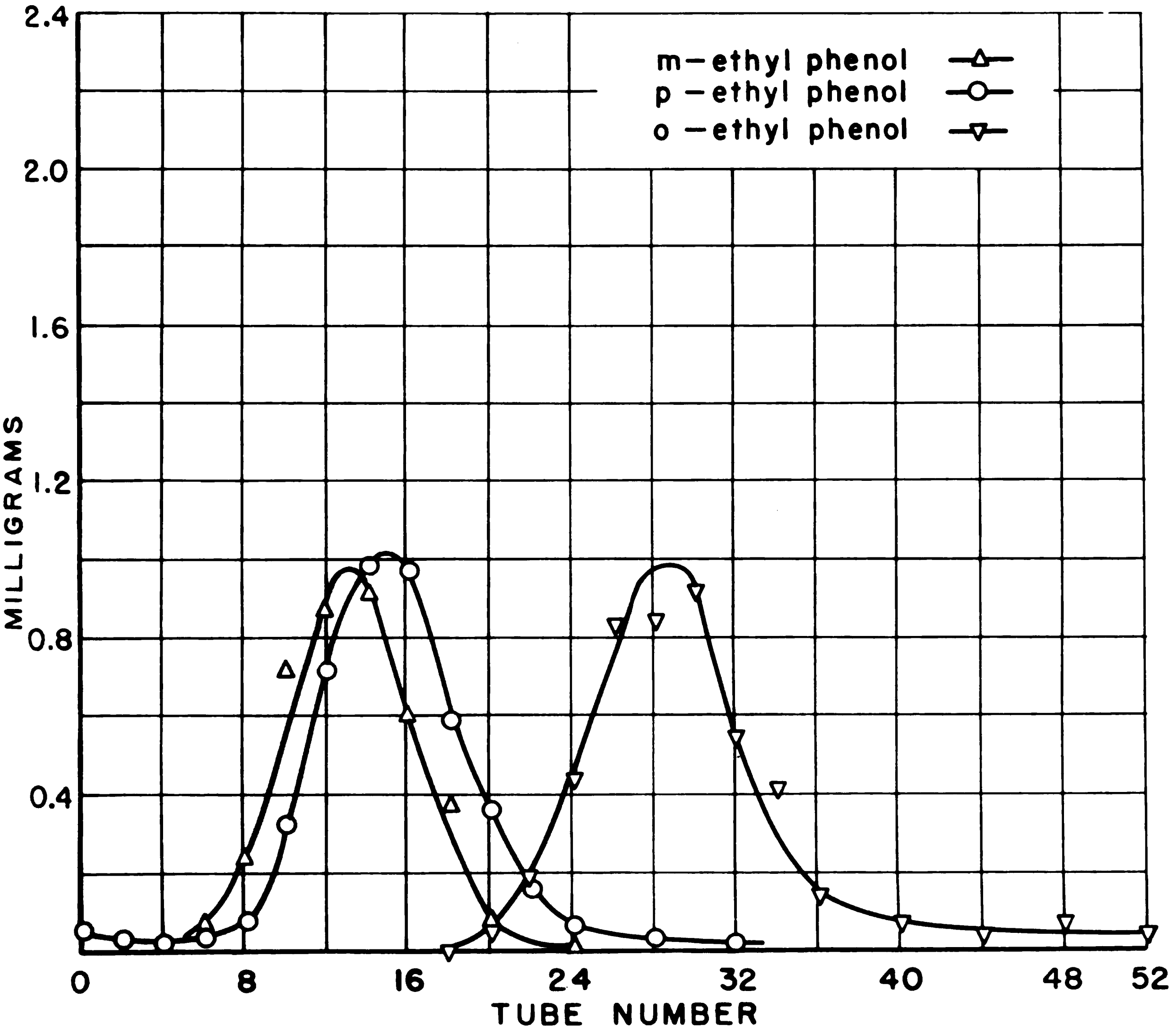

Figure 27. - Separation of ethyl phenols: System, cyclohexane/phosphate buffer pH 11.08; sample, 10 mg. each.

ortho-, meta-, and para-ethylphenols, shows[96] that 3,5-xylenol and ortho-ethylphenol can be readily separated from their respective mixtures. The shapes of the distribution curves (figs. 26 and 27) indicate that each compound distributes itself essentially independently of the others in accordance with the distribution law.

A study of the partition coefficients of dicycle phenols[97] demonstrated that the relative steric influence of methylene groups in six-membered rings is greater than that of five-membered rings. 4- and 5-indanol were isolated from the phenolic mixture obtained in the hydrogenation of Pittsburgh-bed (Bruceton) coal.[98]

The partition coefficients of 18 polynuclear compounds were also determined.[99] Partial saturation of ring structures and introduction of alkyl side chains increased the partition coefficients of parent compounds. Ring systems containing hetero nitrogen and oxygen atoms have lower partition coefficients than the corresponding carbocyclic structures. These effects permit application of the countercurrent distribution method to separation and analysis of polynuclear compounds. The countercurrent distributions of phenanthrene, acenaphthene, and a mixture of carbazole homologues were performed as typical examples. The countercurrent distribution technique permits the measurement of partition coefficients of the components of a mixture without isolating the individual compounds. These coefficients should be considered in the same category as the boiling point, melting point, and refractive index of a compound.

When the middle oils obtained from the hydrogenation of coal were hydrogenated at 450° and 475° C. in a stainless-steel autoclave in the absence of any added catalyst, very little of the phenolic material was converted to neutral products.[1] When the phenolic fraction of the middle oil was isolated prior to hydrogenolysis and then treated, more conversion of high-boiling to low-boiling phenols occurred, but this was accompanied by oxygen elimination and the production of about 20 percent neutral material.

Characterization of Coal

The almost complete lack of knowledge of the chemical composition of coal in the past has greatly hindered the development of improved coal-hydrogenation processes. Earlier work has shown, however, that many of the polynuclear hydrocarbons found in coal-hydrogenation oils may be explained in part by cyclodehydrogenation of less complex hydrocarbons. The attack on this problem was continued by aromatic cyclodehydrogenation[2] and auxiliary related studies.[3][4][5]

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[96] Golumbic, C., Partition Studies on Phenols. II. Separation of Esomeric Phenols by Countercurrent Distribution: Jour. Am. Chem. Soc., vol. 71, August 1949, pp. 2627-2629.

[97] Orchin, M., and Golumbic, C., Partition Studies on Phenols. III. Steric Effects: Jour. Am. Chem. Soc., vol. 71, December 1949, pp. 4151-4152.

[98] Golumbic, C., Woolfolk, E. O., Friedel, R. A., and Orchin, M., Partition Studies on Phenols. IV. Isolation of Indanols from Coal-Hydrogenation Oils: Jour. Am. Chem. Soc., vol. 72, May 1950, pp. 1939-1942.

[99] Golumbic, C., Separation and Analysis of Polynuclear Compounds by Countercurrent Distribution: Anal. Chem., vol. 22, April 1950, pp. 579-582.

[1] Orchin, M., and Storch, H. H., Hydrogenolysis of Phenols in Middle Oils from Coal Hydrogenation: Jour. Soc. Chem. Ind. (London), vol. 69, April 1950, pp. 121-122.

Research on the synthesis of fluoranthene and its derivatives led to the discovery of a method of production through the cyclohydrogenation of phenylnapthalene or derivatives thereof.[6] Fluoranthene is important in the dye industry where it is used for the preparation of certain intermediates. This method permits the use of readily available starting materials from which fluoranthene can be synthesized in a single direct step.

Examination of dispersions of a bituminous coal in phenanthrene by X-ray-powder photography revealed a characteristic diffraction pattern which was found to be identical with that given by partly amorphous abietic acid and aluminum and ferric abietates.[7] No reliable estimate of the concentrations of these constituents could be made, but qualitative observations indicated that they are minor constituents.

A German manuscript describing an investigation of the synthesis of branched-chain hydrocarbons in the laboratories of the Kaiser Wilhelm Institute for Coal Research was translated and published.[8] The paper contains data on the development of the catalyst for the isosynthesis, on the effect of temperature and pressure of operation, and on the composition of the products of the reaction.

LIQUEFACTION OF COAL BY HYDROGENATION OF CARBON MONOXIDE: RESEARCH AND DEVELOPMENT

Reaction Mechanism Studies

Metallic Carbides: The Magnetic Balance

A new method of synthesizing cementite (Fe_3C) that provides a good yield of product, not highly sintered or contaminated with free carbon, was found.[9]

The magnetic balance (fig. 28) makes it possible under certain circumstances to follow quantitatively the course of an isothermal reaction in the solid phase,

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[2] Orchin, M., Reggel, L., and Friedel, R. A., Aromatic Cyclodehydrogenation. IX. Further Studies on Ring Oxygen Compounds: Jour. Am. Chem. Soc., vol. 71, August 1949, pp. 2743-2746.

[3] Orchin, M. and Friedel, R. A., Structure of the Benzfluorenes and Benzfluorenones: Jour. Am. Chem. Soc., vol. 71, September 1949, pp. 3002-3005.

[4] Orchin, M., Synthesis of Perylene: U. S. Patent 2,478,205, Aug. 9, 1949.

[5] Orchin, M., and Wender, I., Apparatus for Measuring Gas Absorption or Evolution during Organic Reactions: Anal. Chem., vol. 21, July 1949, pp. 875-876.

[6] Orchin, M., and Reggel, Leslie, Synthesis of Fluoranthene and Its Derivatives: U. S. Patent 2,503,292, Apr. 11, 1950.

[7] Golumbic, C., Hofer, L. J. E., Peebles, W. C., and Orchin, M., Detection of Abietic Acid in Bituminous Coal: Jour. Soc. Chem. Ind. (London), vol. 69, April 1950, pp. 100-102.

[8] Pichler, Helmut, and Ziesecke, Karl-Heinz (translated by Brinkley, R., technical revision by Golumbic, N.), The Isosynthesis: Bureau of Mines Bull. 488, 1950, 39 pp.

[9] Hofer, L. J. E., and Cohn, E. M., Synthesis of Cementite: Jour. Chem. Phys., vol. 18, May 1950, pp. 766-767.

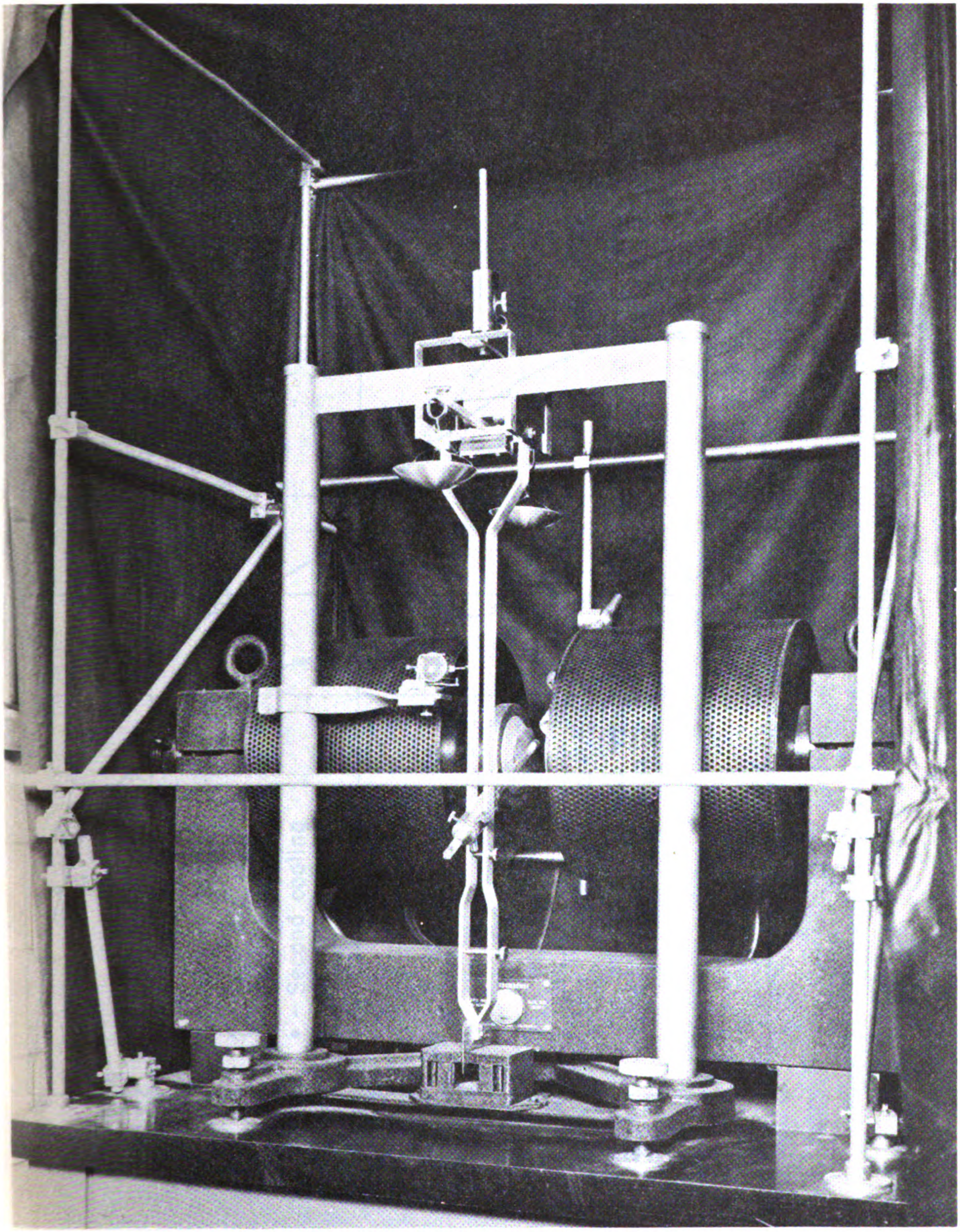

Figure 28. - Magnetic balance.

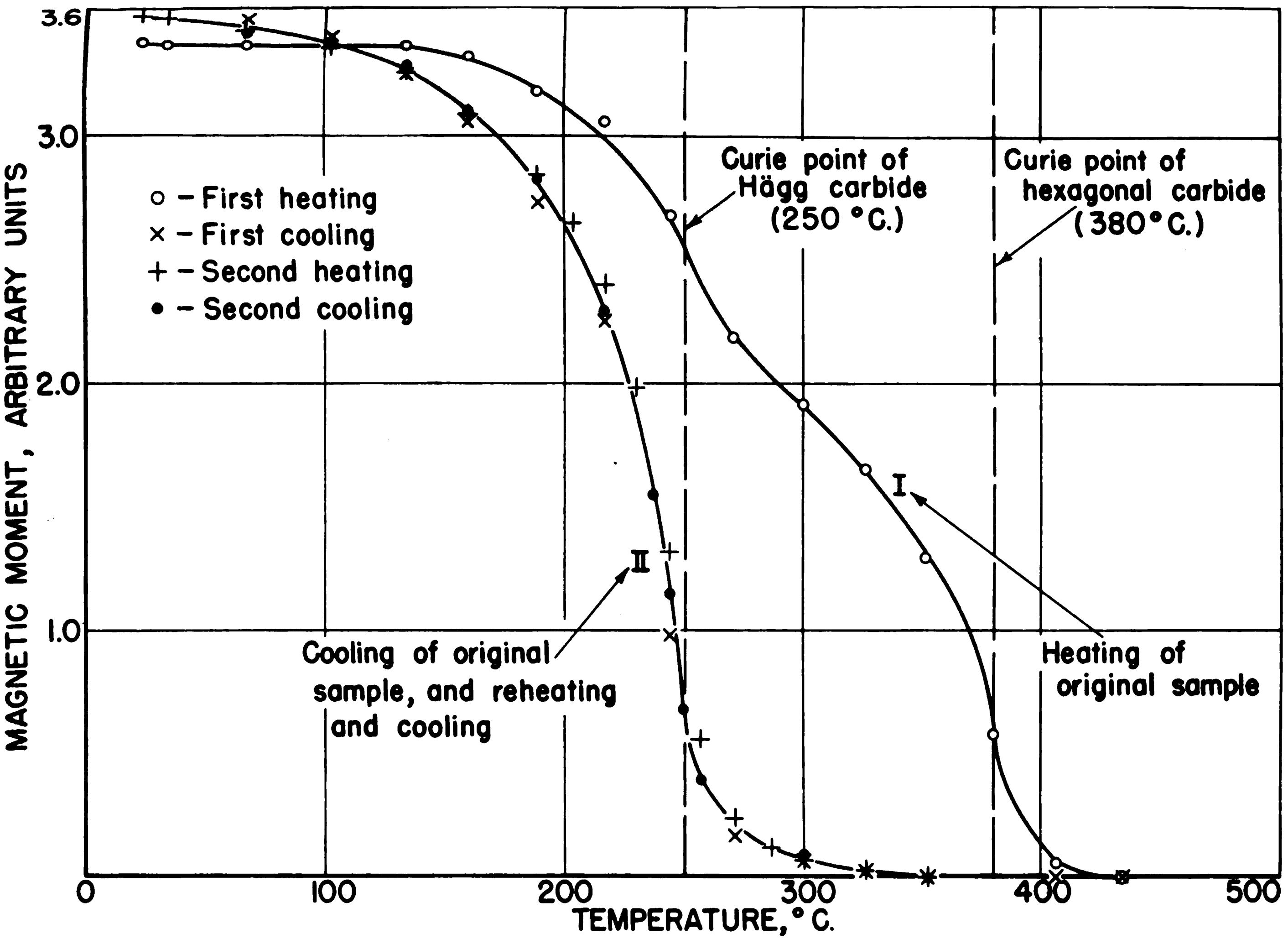

Figure 29. - Thermomagnetic curves showing the presence of the Hägg and hexagonal iron carbides in copper-promoted Fischer-Tropsch catalyst and the reversibility of the Hägg carbide curve.

by observing the change of the force exerted by a magnetic field on a sample that undergoes a chemical and simultaneous ferromagnetic change. This continuous physical analysis has been found to be especially useful in the case where a weak magnetic substance is converted to a strong ferromagnetic one (under the experimental conditions).[10] This method was invaluable in coordinating the widely scattered data for the various iron carbides, as reported by many investigators who carried out their studies along different lines.[11] Two of the properties obtained in this manner, the Curie point and the specific magnetization, can be determined from the thermomagnetic curve, a typical example of which is shown in figure 29. The specific magnetization is a characteristic property of a ferromagnetic phase, which decreases with increasing temperature and disappears at a temperature characteristic of that phase. This temperature is called the "transition temperature" or "Curie point." For practical purposes, the Curie point is usually defined as the point of inflection of a specific magnetization-temperature plot. Thus, the Curie point serves for qualitative identification, while knowledge of the specific magnetization makes quantitative estimates possible. Quantitative determination must be made at high field strengths where the ferromagnetic phases are magnetically saturated. High field strengths are also desirable for following reactions at constant temperature as a function of time. Besides the usual kinetic information, such studies can reveal modes of phase transition (continuous or discontinuous) which may not be readily accessible by any other means, and the method is suitable for following reactions in the solid state (where no changes in gas pressure occur) continuously and without interruption.

In applying this analytical procedure to the study of the isothermal decomposition of cobalt carbide, the rate of decomposition of cobalt carbide was measured by the change of magnetic force experienced by a decomposing sample between 300° and 360° C. in a constant magnetic field of constant field gradient. The measured values were then converted to percent cobalt carbide with the following two assumptions: (1) The initial magnetization of the sample was due to free cobalt, and (2) the force was directly proportional to the amount of cobalt present. The results showed that, in the range from about 80 to 25 percent cobalt carbide, the decomposition-time curves were linear, and hence the reaction was of apparent zero order in that range.

Reduced cobalt-thoria-kieselguhr catalysts produced increasing amounts of free carbon and decreasing amounts of cobalt carbide on carburization with carbon monoxide at increasing temperatures in the range 243° to 298° C.[12] At the high temperatures, the maximum amount of carbide is formed within the first 5 hours of carburization. Free carbon tends to make the catalyst inaccessible to further carbide formation.

Reaction-Rate Studies

The rate of synthesis of hydrocarbons on cobalt Fischer-Tropsch catalysts is dependent upon temperatures, operating pressure, composition, and flow of synthesis gas, as well as the chemical and physical nature of the catalyst. The principal primary reaction of the synthesis on cobalt catalysts may be represented by the equation,

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[10] Hofer, L. J. E., and Cohn, E. M., Studies of Ferromagnetic Materials: Cons. Eng. Corp. Recordings, vol. 4, March 1950, p. 5.

[11] Pichler, H., and Merkel, H. (translated by Brinkley, Ruth, with preface and foreword by Hofer, L. J. E.), Chemical and Thermomagnetic Studies on Iron Catalysts for Synthesis of Hydrocarbons: Bureau of Mines Tech. Paper 718, 1949, 108 pp.

[12] Hofer, L. J. E., Peebles, W. C., and Bean E. H., X-Ray Diffraction Studies of the Action of Carbon Monoxide on Cobalt-Thoria-Kieselguhr Catalysts. II: Jour. Am. Chem. Soc., vol. 72, June 1950, pp. 2698-2701.

$$2H_2 + CO \longrightarrow 1/n\ (CH_2)_n + H_2O,$$

the usage ratio of hydrogen to carbon monoxide being about 2:1 over a wide range of gas compositions. Previous work demonstrated that bulk-phase cobalt carbide was not formed in the synthesis and that its presence in large amounts seriously decreased the catalytic activity.

A study of the differential reaction rates involved in Fischer-Tropsch synthesis, with a cobalt catalyst, was undertaken,13/ in which the synthesis rate and the rates of formation of methane and carbon dioxide were investigated as a function of bed length. Cobalt-thoria-kieselguhr catalyst was operated at atmospheric pressure, with hydrogen to carbon monoxide ratios of 3.5:1, 2:1, and 0.9:1, using argon as an inert reference component. The synthesis rate was found to be high in the initial part of the bed and lower and nearly constant throughout a large portion of the catalyst bed, until the reactants were fairly completely consumed. The data suggest that the rate-controlling step is some process at the catalyst surface and not the rate of adsorption of reactants, which would be strongly dependent upon the partial pressures of the reactants, a characteristic which was not substantiated by the experimental data. A mechanism in which desorption of products is the rate-determining step predicts, qualitatively, many of the characteristics of the synthesis. The reaction is very complicated, because the catalyst is heavily covered with the hydrocarbon products, and differences in the nature of the catalyst surface may be a function of the composition of the gas in contact with catalyst. Over the range of relatively constant rate of synthesis, the gas composition varied considerably.

The ratio of saturated to unsaturated hydrocarbons present at any point in the catalyst bed varied with the ratio of hydrogen to carbon monoxide in the gas at that part of the bed. This indicates that olefins may be a primary product of the reaction, and that these olefins may then be hydrogenated or undergo further reaction to form hydrocarbons of higher molecular weights at the point of formation or in later parts of the catalyst bed.

The processes that form methane appear to be functions of the partial pressures of hydrogen and hydrocarbons, and processes forming carbon dioxide appear to be functions of the partial pressures of water vapor and carbon monoxide. The formation of both methane and carbon dioxide increased with increasing temperature. These reactions occur, at least, to some extent over the entire catalyst bed.

Effect of Reduction on Metal Oxide Catalysts

Changes that occur during reduction may be divided into three classes: (1) Sintering of the unreduced material may occur at the reduction temperature, the "glowing" of oxide gels being an extreme example of this phenomenon; (2) the transition of oxide to metal may be accomplished by a sizeable decrease in volume; and (3) reorientation of the reduced metal into a stable form takes place. If considerable reorientation of the freshly reduced metal does not occur simultaneously with the reduction, the change in volume should make the metal-to-oxide interface accessible

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13/ Anderson, R. B., Krieg, A., Friedel, R. A., and Mason, L. S., Fischer-Tropsch Synthesis - Differential Reaction Rate Studies with Cobalt Catalyst: Ind. Eng. Chem., vol. 41, October 1949, pp. 2189-2197.

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to the reducing gas. A study of these changes in the physical structure of precipitated cobalt and iron oxide catalysts on reduction was completed with the electron microscope.[14] To obtain further information about changes occurring during reduction, a number of metal oxides were also studied. A secondary purpose of this research was to determine the usefulness of the electron microscope in studies of granular catalysts.

Consideration of the electron-microscope studies of catalysts indicates that these are useful and informative but that the micrographs are seldom simple to interpret. It is desirable to consider the micrographs, with surface-area results from gas-adsorption studies. From a comparison of particle size and shape with surface area, some estimate of the porosity of the particle can be made. Some precipitated metal oxides have gel structures, with fairly high mechanical strength and high surface areas. Some gels have been postulated to be "felts" of tiny filaments. The electron micrographs support this postulate. Fused-metal oxide catalysts represent an opposite extreme to metal oxide gels. Fused catalysts, such as those used in the ammonia synthesis, are dense solids, with very little internal surface area. On reduction, these catalysts form a porous structure, but their external (unreduced) volumes are not changed. The surface areas of the precipitated oxides and catalysts studied always decreased on reduction.

Separation and Identification of Products of Hydrogenation of Carbon Monoxide

The composition of synthetic liquid fuels obtained from the Fischer-Tropsch process is of interest from the viewpoint of reaction mechanism and in determining the applicability of the products as fuels. Except for general product-distribution studies, little quantitative work on this problem had been reported. A study of the products obtained from laboratory-scale catalyst testing units was therefor undertaken.[15] In the case of iron catalysts, where considerable carbon dioxide is found in the exit gas, studies such as these were facilitated by the development of an improved caustic scrubber.[16]

The product distribution from precipitated cobalt catalysts was obtained by fractional distillation of the liquids and mass spectrometer analysis of the liquids and gases. In connection with the evaluation of distillation columns operating at reduced pressures, the use of a test mixture of a hydrocarbon whose hydrogen atoms are of mass one and the same hydrocarbon containing some deuterium atoms (hydrogen atoms of mass two) is highly desirable. Accordingly, a method of analysis for hydrocarbons containing deuterium was developed,[17][18] which is superior to the analytical methods previously used. In a similar manner, the development of a simple method for introducing liquids into a mass spectrometer and the study of negative peaks obtained in mass spectra were extremely valuable.[19/][20/]

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[14] McCartney, J. T., Seligman, B., Hall, W. K., and Anderson, R. B., An Electron-Microscopic Study of Metal Oxides and Metal Oxide Catalysts: Jour. Phys. Colloid Chem., vol. 54, April 1950, pp. 505-519.

[15] Friedel, R. A., and Anderson, R. B., Composition of Synthetic Liquid Fuels. I. Product Distribution and Analysis of C_5-C_8 Paraffin Isomers from Cobalt Catalyst: Jour. Am. Chem. Soc., vol. 72, March 1950, pp. 1212-1215; correction, May 1950, p. 2307.

[16] Krieg, A., Dudash, A. P., and Anderson, R. B., Improved Caustic Scrubber: Ind. Eng. Chem., vol. 41, July 1949, p. 1508.

[17] Orchin, M., Wender, I., and Friedel, R. A., Determination of Deuterium in Water-Conversion to Methyl Deuteride and Methane and Measurement by Mass Spectrometer: Anal. Chem., vol. 21, September 1949, pp. 1072-1073.

[18] Friedel, R. A., and Sharkey, A. G., Mass Spectrum of Hydrogen Deuteride (HD): Jour. Chem. Phys., vol. 17, June 1949, pp. 584-585.

The results indicated a high yield of methane, followed by a minimum at C_2, a broad maximum from C_5 to C_{11}, and a gradual decrease to C_{20}. An analysis of paraffin isomers was made in the C_5-C_8 range. Of particular interest was the slight, but progressive, decrease of straight-chain isomers, the major components, with molecular weight. Calculated values for ethyl- and dimethyl-derivatives were very close to zero in all cases, being either slightly positive or negative. This is considered valid evidence for the absence of appreciable concentrations, but the difficulties of carrying out separations on a small scale did not allow precluding the presence of traces. No indication of any compound containing a quaternary carbon atom has been found. These experimental results on isomer distribution of pentane to octane were also deduced from probability considerations.[21/][22/]

Study by means of the infrared spectrometer of the C_6 to C_8 olefins in the product showed the predominance (64-82 percent) of olefins, with the double bond in positions other than the alpha (1,2) position, and that the proportion of these olefins in the product increased with its molecular weight. The product from iron catalysts, on the other hand, contained as high as 90 percent alpha olefins in the C_6 range. Analyses of Diesel oil and wax fractions from iron catalysts in the internally cooled converter have shown, however, that olefins other than alpha predominate in these fractions by about 7 to 1. Since olefins other than alpha are favored thermodynamically, the approach toward equilibrium due to longer residence in the converter, which results from the current practice of recycling the heavy products, would increase the concentration of these olefins at the expense of the alpha olefins.

Homologation of Alcohols

Because of the presence of relatively large amounts of alcohols in the products from the gas synthesis, a study is in progress on possible mechanisms for their formation. The conversion of an alcohol into the primary alcohol containing one carbon atom more than the original is usually a tedious procedure. Recently, a simple one-step homologation reaction was developed,[23/] which consists of treating the alcohol with synthesis gas in the presence of a cobalt catalyst under conditions resembling those employed in the Oxo reaction.

The Oxo or hydroformylation reaction employs the conversion, by means of synthesis gas, of an olefin to a mixture of aldehydes containing one carbon atom more than the starting material. The aldehydes are then, customarily, reduced in a separate step, or they may be converted directly to the alcohol by operating the usual Oxo reaction

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[19/] Friedel, R. A., Sharkey, A. G., Jr., and Humbert, C. R., Liquid Sampling for Analysis by Mass Spectrometer: Anal. Chem., vol. 21, December 1949, pp. 1572-1573.

[20/] Sharkey, A. G., and Friedel, R. A., Anomalous Negative Peaks in Mass Spectra: Jour. Chem. Phys., vol. 17, October 1949, p. 998.

[21/] Weller, S., and Friedel, R. A., Isomer Distribution in Hydrocarbons from the Fischer-Tropsch Process: Jour. Chem. Phys., vol. 17, September 1949, pp. 801-803.

[22/] Weller, S., and Friedel, R. A., Isomer Distribution in Fischer-Tropsch Hydrocarbons: Jour. Chem. Phys., vol. 18, January 1950, pp. 157-158.

[23/] Wender, I., Levine, R., and Orchin, M., Homologation of Alcohols: Jour. Am. Chem. Soc., vol. 71, December 1949, pp. 4160-4161.

at slightly higher temperatures. The homologation reaction, however, need not proceed via an olefin intermediate followed by hydroformylation-hydrogenation, as shown by the fact that β-phenylethyl alcohol is obtained from benzyl alcohol according to the reaction:

$$PhCH_2OH + 2H_2 + CO \longrightarrow PhCH_2CH_2OH.$$

Similarly, isobutyl alcohol was obtained from isopropyl alcohol and isoamyl alcohol from tertiary butyl alcohol.

Study of Fundamental Properties of Fixed and Fluidized Beds

The pressure drop in gases and liquids flowing through beds of granular material is an important factor in the engineering of absorption towers, catalytic reactors, and filters. Recent investigations into the fundamentals of fluidization of solids have shown the intimate relation between pressure drop and fluidization phenomena. It has been established that, if upward flow of a fluid through a bed of granular material is gradually increased, the solids will begin to fluidize when the pressure drop across the bed equals the buoyant weight of the solids. In order to apply this relationship more conveniently to practical examples, a nomogram was developed which facilitates a comparison of pressure drop with fluid velocity, fluid viscosity, size and shape of packing particles, and void space in the bed.24/

Another problem of fundamental importance is that involving heat flow. Although considerable work has been reported on various types of fixed and moving beds and on a system characterized by clouds of fine particles, little information was available on heat transmission through fluidized beds. A study of this problem25/ resulted in the development of three correlations applicable to particles in the range 0.0015 to 0.0045 inch. With air, carbon dioxide, or helium under various operating conditions, coefficients as low as 0.25 and as high as 8 B.t.u./(hour) (°F.) (square feet) were observed. Within the limits of the experimental data, it was shown that the bed height, the concentration of solids in the bed (state of expansion), the density of the solid phase, and the vessel diameter had no effect upon heat transfer. Effect of slugging upon heat transfer was found to be negligible. Channeling, however, had a very pronounced effect, which was reflected by the erratic temperature distribution through the bed. Absolute particle size and shape are considerably more important than size distribution in inducing channeling.

Pilot-Plant Synthesis of Hydrocarbons from Carbon Monoxide and Hydrogen

Work was continued in pilot-plant units using the internally cooled, moving-bed converter design. In this system, the catalyst is cooled by a flow of circulating oil, which also lifts the catalyst particles and keeps them suspended in a stream of liquid and gas. In most of the pilot plant runs a fused-synthetic-ammonia-type catalyst was reduced with hydrogen at about 450° C. before use in the synthesis. The gases present in the converter were found to be increasingly soluble in the circulating oil in the following order: Hydrogen, carbon monoxide, and carbon dioxide. A conversion about 65 percent as great was obtained by introducing the synthesis gas into the free space at the top of the converter as was obtained by bubbling the gas

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24/ Weintraub, M., and Leva, M., Pressure Drop Through Fixed and Fluidized Beds - A Nomogram: Chem. Eng., vol. 47, January 1950, pp. 110-113.

25/ Leva, M., Weintraub, M., and Grummer, M., Heat Transmission Through Fluidized Beds of Fine Particles: Chem. Eng. Progress, vol. 45, September 1949, pp. 563-572.

up through the converter in the conventional method. This experiment shows that diffusion of the reactants to the catalyst surface is not the rate-controlling step in the process.

Current pilot-plant operations are being directed toward determining the conditions for minimum production of gaseous hydrocarbons. A comparison of Alumina and Cyclocel bauxite catalysts, as a means of removing oxygenated compounds from the overhead vapors, showed both materials to be effective in this respect. Cyclocel bauxite, however, produced appreciable shifting of the double bond, which was not observed with Alumina bauxite.

A stripper column was operated in a manner to simulate actual operation in series with the internally cooled converter. In this column, the dissolved product water was separated from the circulating oil by countercurrent flow of the oil and the feed and recycle gases. As a result, the H_2:CO usage ratio was increased, because the water-gas shift reaction in the converter was depressed.

By scrubbing the carbon dioxide from the recycle gas with potassium carbonate solution during gas-recycle operation, 90-percent conversion of synthesis gas was possible in a single stage by increasing the normal reaction temperature about 10° (to 260° C.). Without removal of carbon dioxide from the recycle gas, not more than 85 percent synthesis-gas conversion could be obtained in a single stage, even at temperatures up to 295° C. and at higher gas-recycle rates.

SYNTHETIC LIQUID FUELS DEMONSTRATION PLANTS

The Coal-Hydrogenation Demonstration Plant went through the necessary break-in procedures. During the year the liquid-phase unit made an exploratory tar oil run of 2 months' duration and three coal runs, during which 150,000 gallons of tar oil was hydrogenated and 1,300 tons of bituminous coal was liquefied.

The Gas-Synthesis Demonstration Plant construction is practically complete. The 1-ton-per-day capacity oxygen-production unit is operating on routine basis. The atmospheric pulverized-coal gasifier made numerous short exploratory runs and an extended run of 2 weeks' duration. The other portions of the plant are ready for trouble shooting and break-in runs.

The emphasis of the engineering work is definitely shifting from the design and construction stages to operational problems, cost estimates, and commercial evaluation of the different processes.

Coal-Hydrogenation Plant

Two papers[26][27] described both the process and special features of construction of the demonstration plant.

A successful run was made in the vapor-phase section of the plant.[28] Light petroleum distillate was hydrogenated to motor gasoline, and a distillate obtained from the tar oil, produced by low-temperature carbonization of North Dakota lignite, was converted to a high cetane number Diesel fuel oil and motor gasoline.

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[26] Markovits, J. A., The Coal-Hydrogenation Demonstration Plant at Louisiana, Mo.: Trans. Am. Soc. Mech. Eng., vol. 72, May 1950, pp. 349-355 (Paper 49 - PET-3).

[27] Hirst, L. L., Complete Coal-to-Oil Demonstration Plants: Ind. and Power, vol. 57, August 1949, pp. 90-93, 116.

[28] Clarke, E. A., Chaffee, C. C., and Hirst, L. L., Hydrogenation of Petroleum and Lignite Tar Distillates: Bureau of Mines Rept. of Investigations 4676, 1950, 27 pp.

Following a low-pressure (1,600-pounds-per-square-inch) "dummy" run with nitrogen to give the operators experience and to make a break-in test of the equipment, the system was pressured to 10,300-pounds-per-square-inch gage with hydrogen. The temperature was raised slowly to 835° F. and held there to activate the catalyst in the vapor-phase converter. After activation of the catalyst, the temperature was reduced, and a vapor-phase hydrogenation break-in run was made using a 43° A.P.I. gasoline-naphtha-gas-oil blend prepared from an Oklahoma City crude petroleum oil. The charge during the petroleum-hydrogenation run was made up of a blend of 11 percent of 61° A.P.I. straight-run gasoline, 31 percent of 47° A.P.I. naphtha, and 58 percent of 39° A.P.I. gas oil. A total of 29,360 gallons of this blend, with 2,960 M cubic feet of 95 percent hydrogen make-up gas was charged during the 4-day run. Although operating conditions were varied to gain information concerning the operation of controls and the product, the average run conditions were as follows:

	P.s.i.g.
Pressure	
Converter inlet	9,750
Hydrogen feed	9,975
Recycle compressor suction	9,715
Recycle discharge	10,000
	°F.
Temperature	
Preheater inlet	395
Preheater outlet	721
Catalyst beds	699
Cold catchpot	92

Hydrogenation at temperatures above 720° F. produced a light product higher than 70° A.P.I. The distillation unit was put on stream to fractionate the hydrogenated product into its components.

The products and yields obtained were:

	°A.P.I.	Gallons	M c.f.
Gasoline	66	15,725	-
Wash oil (naphtha)	48	5,840	-
Bottoms	43	4,280	-
Off gases	-	-	975

Distillation curves showed that the quantity distilling over 400° F. was increased from 25 percent in the feed to 65 percent in the hydrogenated product, in one pass through the converter. The gasoline cut had an octane number of 63 by the A.S.T.M. D357-48 C.F.R. motor method.

The vapor-phase hydrogenation feed stock then was changed to a lignite-tar distillate oil in order to produce a specification Diesel fuel. This stock was prepared from a 10° A.P.I. North Dakota lignite-tar oil by removing the low-boiling portion and the asphaltic material unsuitable for hydrogenation over the Welheim K-536 catalyst. The portion used represented 70 percent of the raw oil and had a gravity of 12° A.P.I.

The lignite-tar distillate was charged to the unit without any purging of petroleum oil. The total charge, including the petroleum in the system was made up as follows:

Lignite-tar distillate	gallons	12,225
Petroleum oils in system	do.	5,480
Hydrogen make-up gas	M c.f.	2,930

Although temperatures were varied during the run to obtain the optimum reduction of tar acids, the average operating conditions were as follows:

Pressure	P.s.i.g.
Converter inlet	9,725
Hydrogen feed	10,550
Recycle compressor suction	9,690
Recycle compressor discharge	10,100

Temperature	°F.
Preheater inlet	390
Preheater outlet	730
Catalyst beds	727
Cold catchpot	91

The products and yields obtained were:

Gasoline	gallons	8,835
Wash oil (naphtha)	do.	615
Diesel fuel oil	do.	4,520
Slop oil	do.	520
Off gases	M c.f.	955

Distillation curves showed that the quantity distilling over 400° F. was increased in one pass by 25 percent as compared to 40 percent for the petroleum. The gasoline had an octane number of 65 by the motor method. This gasoline has given satisfactory service at this station in all types of motor vehicles formerly using regular motor-grade gasoline.

The bottoms from the product distillation were finished for Diesel fuel oil. Table 16 is a comparison of the properties of the Diesel fuel oil produced with the specifications used by one of the large western railroads for the purchase of Diesel fuel from petroleum:

TABLE 16. - Comparison of Diesel fuel oil produced by coal hydrogenation with railroad specifications

		Louisiana test run	Railroad specifications
Gravity	A.P.I.	38.5	32 - 40
Boiling range:			
10 percent	°F.	440	425 - 500
90 percent	°F.	578	540 - 620
End point	°F.	644	675 max.
Recovery	percent	98	98
Viscosity at 100° F.	S.S.U.	33.5	35 - 45
Flash point (PM)	°F.	194	150 min.
Pour point	°F.	10	5
Ash	percent	0.002	0.1 max.
Cetane number		56	50 min.
Carbon residue (10 percent bottoms)	percent	0.06	0.25 max.

This fuel was used successfully on a 200-mile trial run in a Diesel-electric locomotive hauling a loaded eight-car passenger train.

Four runs have been made on the liquid-phase unit during its first year of operation. The first was an exploratory tar-oil run to test the performance of the equipment and instrumentation. The unit was in continuous operation from October 12 to December 6. During this run, the general reliability of the equipment and its tightness were ascertained. The operators obtained further experience while continuous efforts were being made to have the instruments, particularly control valves, work satisfactorily. The first hydrogenation products appeared at 750° F. The operation was fairly smooth, hydrogenating tar oil at a temperature of 850° F. in the converters, with a 50-50 split between light and heavy oils.

The first coal paste, containing 10 percent of Wyoming Rock Springs coal, was introduced on November 24. The coal content was raised to 25 percent within 20 hours. Operating conditions of pressure, temperature, and flow rates were maintained essentially at the same levels as during the tar-oil operation. The coal-paste phase of the run was continued for 8 days and was terminated when the supply of coal previously obtained to break in the coal-preparation plant was exhausted. The unit was cleaned and inspected, defective instrument tubing was replaced, and improvements in instrumentation were made.

The second liquid-phase hydrogenation run was started on March 31. A light start-up oil blend was used to test the controls and bring the unit up to operating conditions. Coal-tar hydrogenation was started on April 8 and continued until April 12. Operating conditions were fairly smooth.

Coal addition was started to the system on April 12, and the charging rate was rapidly increased to 3,000 pounds per hour, using a paste feed rate of 19 gallons per minute, with gas circulating through the furnace at 240,000 cubic feet per hour. Converter temperatures were held at 840° to 850° F. Shortly after the introduction of coal, instrument failures caused several severe upsets in operations. These conditions were further aggravated by irregular paste injection due to valve troubles. The run was terminated on April 16 because of leaks in the transfer lines between the converters. Inspection of the pumps revealed that all but four of the large injection pumps, as well as several of the smaller pumps, had developed irreparable liquid-end block leaks.

In spite of the pump situation and in view of the delay in obtaining new blocks, it was decided to make a run at 7,500 pounds per square inch to make products, to allow working out more of the mechanical problems, especially those concerned with instruments, and to improve general process control required for smooth operations. After lining out the unit on pasting oil, coal was introduced on May 16. This operation was smooth until converter temperature measurements could not be relied upon. A normal shut-down was made on May 23. Inspection of the equipment showed it to be clean, and for the first time no coke was found in the converters.

For the fourth run, coal-paste injection was started on June 8th and continued for 3 weeks. The operation was the steadiest attained on any run. System pressures, temperatures, rates of flow of liquids and gases, solids removal, uniformity of paste composition, and quality of pasting oil showed a marked improvement, making it possible to charge coal at rates as high as 50 tons per day.

The unit was shut down per schedule on June 27. After cleaning, inspection, and the installation of redesigned fluid-end blocks on the injection pumps, operation will be resumed.

Ammonium molybdate was used as a catalyst when charging coal tar, and stannous oxalate when charging coal.

A total of 140,000 gallons of coal-tar oil and 1,265 tons of coal has been processed during these runs. One hundred sixty-eight thousand gallons of charging stock suitable for further hydrogenation in the vapor phase to make specification motor gasoline have been produced. In addition, there are 90,000 gallons of heavy oils produced during the operations which will require further processing.

A full report of these runs is in preparation and will be published as soon as completed.

Two short runs were made on the steam flash distillation unit for recovering oil from the solids. These operations have shown that:

1. Taking the feed directly from the hot catchpot is not feasible on our unit: (a) It interferes with the operation of the level controls; (b) it is impossible to obtain a steady, measured flow; and (c) the oil-solids mixture is cooled by unavoidable line losses to too low a temperature for flashing.

2. Condensed oil droplets are carried out in the steam exhaust, causing serious hazards.

While these difficulties render the equipment inoperable at present, fortunately, they can be corrected by the installation of a direct-fired heater and a total condenser, which are on order.

Gas-Synthesis Plant

The Gas-Synthesis Demonstration Plant is designed to receive coal by the carload at its gasification unit, to crush and pulverize the coal, and to react it with oxygen and steam to make a synthesis gas. This gas is then to be compressed and purified of sulfur compounds and utilized according to the Fischer-Tropsch synthesis process to make synthetic liquid fuel products, such as gasoline and Diesel oil. The plant capacity is approximately 3,000,000 cubic feet of gas per day, which will be converted into 80 barrels per day of total finished products.

At the end of the fiscal year 1950 construction of this plant is substantially complete. Three of the plant units, namely, the oxygen production and powdered-coal gasification units and the Kerpely gas producer, have been in operation for periods of 2 to 18 months. Two views of the plant are given in figures 30 and 31.

Oxygen Production

The oxygen production unit started initial operations in December 1948 and has been operating since. It produces 24 tons per day of oxygen for use in gasifying coal. Over periods of several months it has been kept in operation 95 percent of the time while producing oxygen of 98-percent purity.

Koppers Powdered-Coal Gasifier

One of the unique units in the demonstration plant is the Koppers-type powdered-coal gasifier. This unit is designed to convert 1 ton of coal per hour to synthesis gas by the use of approximately equivalent quantities of oxygen and steam. The first run at full rates of oxygen, steam, and powdered coal was made in June 1949. Since then 46 runs were made, each run normally of 6 to 10 hours' duration, including 1 run

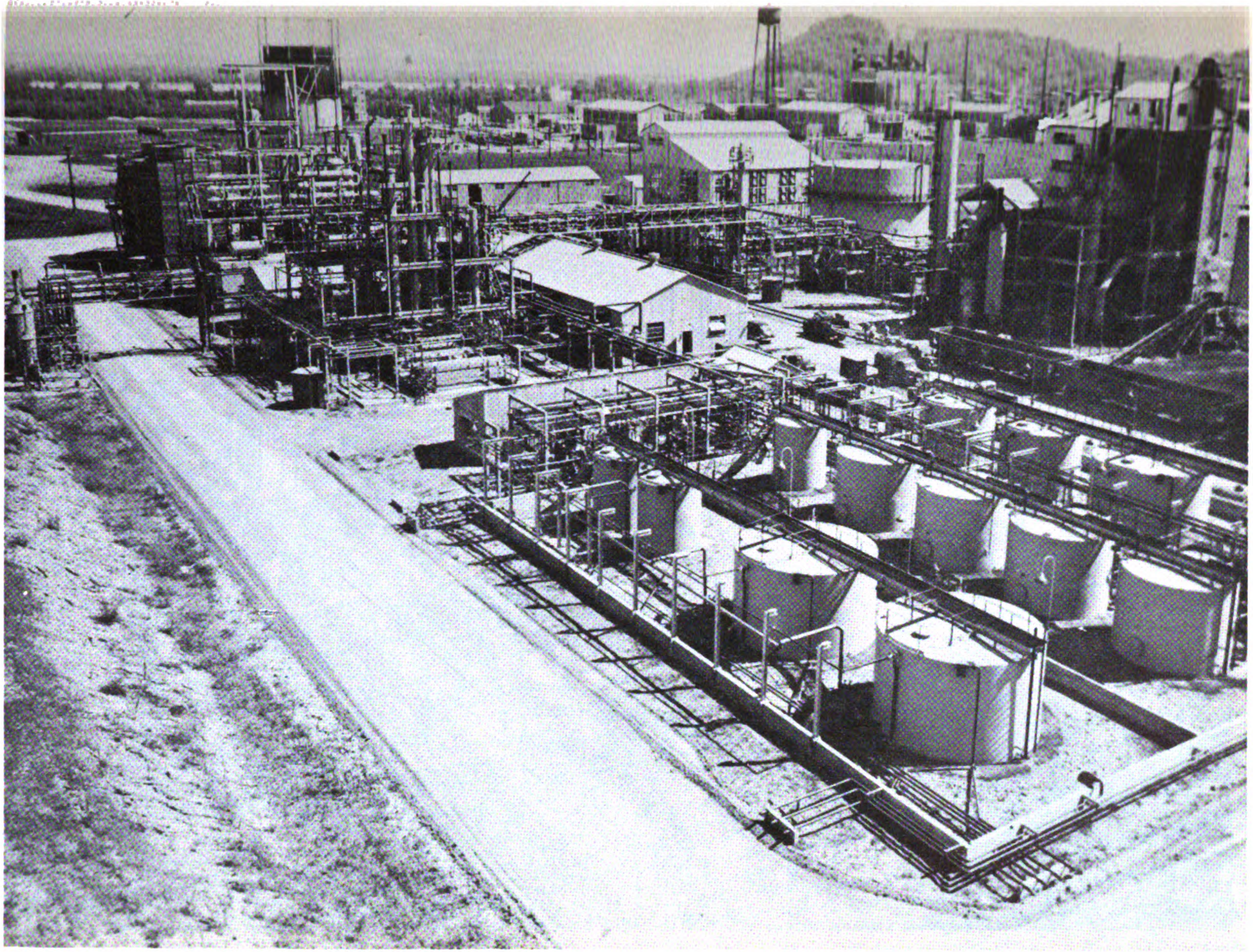

Figure 30. - Gas-Synthesis Demonstration Plant, Louisiana, Mo.

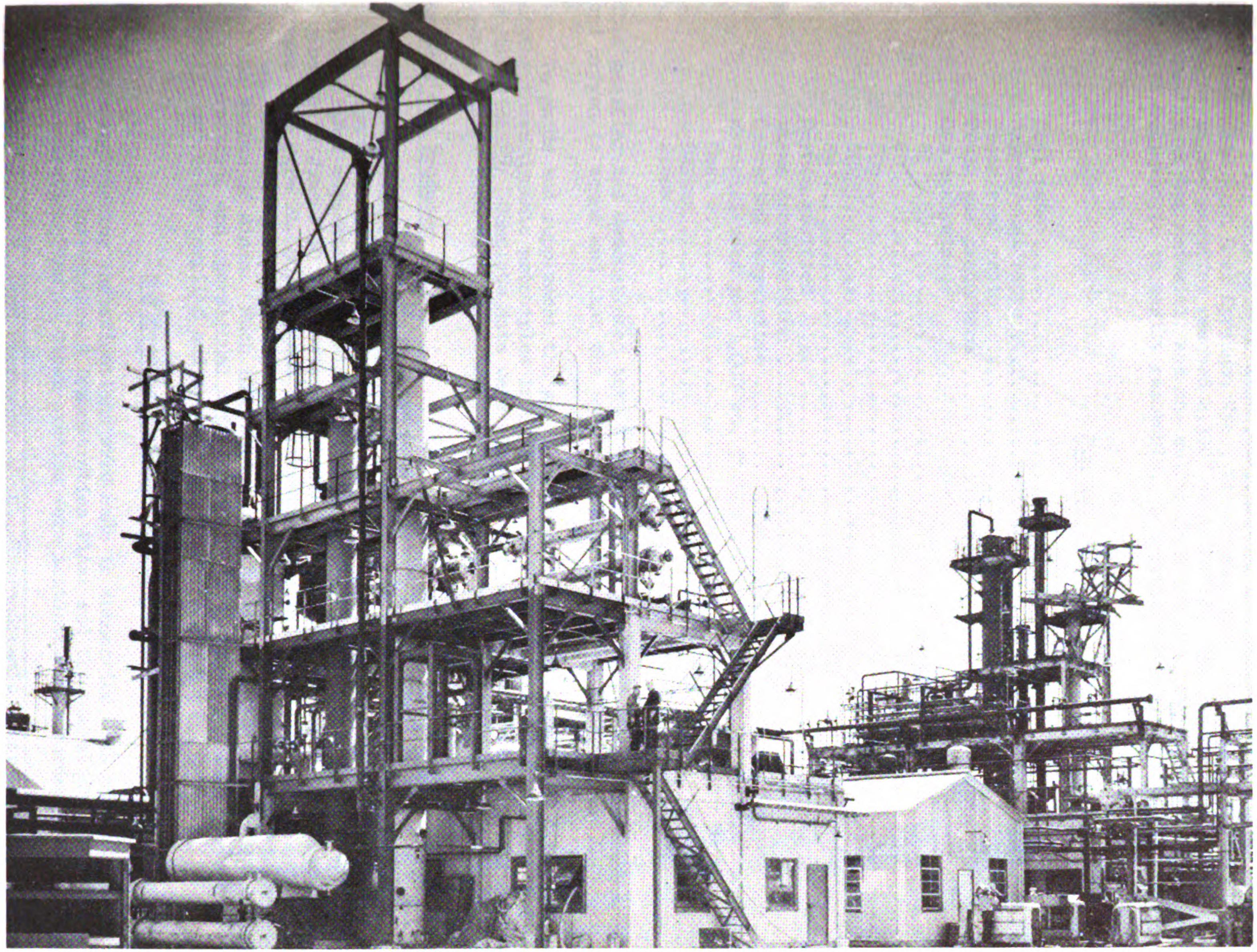

Figure 31. - Gas-Synthesis Demonstration Plant, Louisiana, Mo.

of 11 days. Full data were obtained, calculated, and recorded. All runs to date have been made with Rock Springs, Wyo., bituminous coal. In these trial runs temperatures in the gasifier reached 2,300° to 2,800° F. The produced gas contained practically no tar material, and the sulfur from the coal was converted to a form, which is believed to be easily removable. A typical run yielded the following results:

Run No. 18

Coal rate	lb./hr.	1,976
Oxygen/coal ratio	cu. ft./lb. coal	9.48
Steam/coal ratio	lb./lb. coal	0.91
Total gas production	std. cu. ft./hr.	63,800
Gas analysis:		
H_2	percent	37.8
CO	do.	42.9
CO_2	do.	15.6
CH_4	do.	0.1
N_2	do.	3.3
Carbon gasified	do.	84.2
Steam decomposition	do.	6.7
Operating temperature	°F.	2,300 - 2,600
Material used per 1,000 cu. ft. $CO + H_2$:		
Coal	pounds	38.4
Oxygen	cubic feet	364.0
Steam	pounds	35.0

This gasifier has demonstrated that the principle of making gas from powdered coal and oxygen is sound and that the physical operations are feasible.

To guide and supplement the gasifier operations, a comprehensive study was made of the thermodynamic principles involved in the coal-oxygen-steam reaction and its application.[29] Complete data were also taken from the operation of the circulating pebble heater used to supply steam, super-heated up to 2,300° F., to the gasifier.

Kerpely Gas Producer

The demonstration plant includes a Kerpely gas producer. Normally, these, or similar conventional producers, are operated with coke using air blast. In the interest of higher capacities and high quality of gas, this unit was operated on a continuous basis in June 1950 with oxygen, or with oxygen-enriched air. Concentrations of oxygen used were 21, 50, 75, and 98 percent. Coke was either of pea or nut size. The runs were made under a cooperative agreement between the American Gas Association and the Bureau of Mines. Much information was gained on output capacities, analysis of gas produced, and operational difficulties, limitations, and control, the results of which are being compiled and evaluated.

Engineering Studies and Cost Estimates

Many engineering investigations were made in connection with the design and construction of the Gas-Synthesis Demonstration Plant and the operation of the Hydrogenation Demonstration Plant. Other studies were initiated in accordance with Public

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[29] Batchelder, H. R., and Sternberg, J. C., Thermodynamic Study of Coal Gasification - Applicable to Suspension Gasification of Pulverized Coal: Ind. Eng. Chem., vol. 42, May 1950, pp. 877-882.

Law No. 290 of the 78th Congress and at the request of other government agencies. Some of this work was completed and published.

An index of Technical Oil Mission reels was completed. Additional translations of German documents were made.[30]

At the request of the Corps of Engineers, U. S. Army, a chart of synthetic fuel-plant requirements was prepared. Table 17 is typical of the information shown on the chart for a 10,000-barrel-per-day plant.

TABLE 17. - Requirements for a 10,000-barrel-per-day synthetic fuel plant

	Hydrogenation	Synthine using coal	Synthine using natural gas
Over-all efficiency .... percent	50.5	46.0	52.0
B.t.u./calendar day required	98×10^9	114×10^9	100×10^9
Plant personnel required	1,175	1,045	795
Area required .... acres	70	77	56
Products:			
LP-gas .... bbl./day	2,367	-	-
Gasoline .... bbl./day	7,220	8,290	8,290
Gas oil .... bbl./day	-	1,325	1,325
Fuel oil .... bbl./day	-	385	385
Phenols .... bbl./day	413	-	-
Total bbl./day	10,000	10,000	10,000
Oxygenated compounds .... bbl./day	-	1,104	1,104
Total bbl./day	10,000	11,104	11,104

The water requirements for the three types of synthetic fuel plants, in barrels of water per barrel of products are:

	Hydrogenation	Synthine using coal	Synthine using natural gas
Ample water available (once through operation)	300	390	190
Insufficient water (using cooling towers).	12.7	16.3	10.4
Combination cooling (air and water)	8.75	11.2	7.94

The above figures are for an average coal of approximately subbituminous rank.

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[30] Copies of translations of T.O.M. reels available from the Bureau of Mines, Louisiana, Mo.

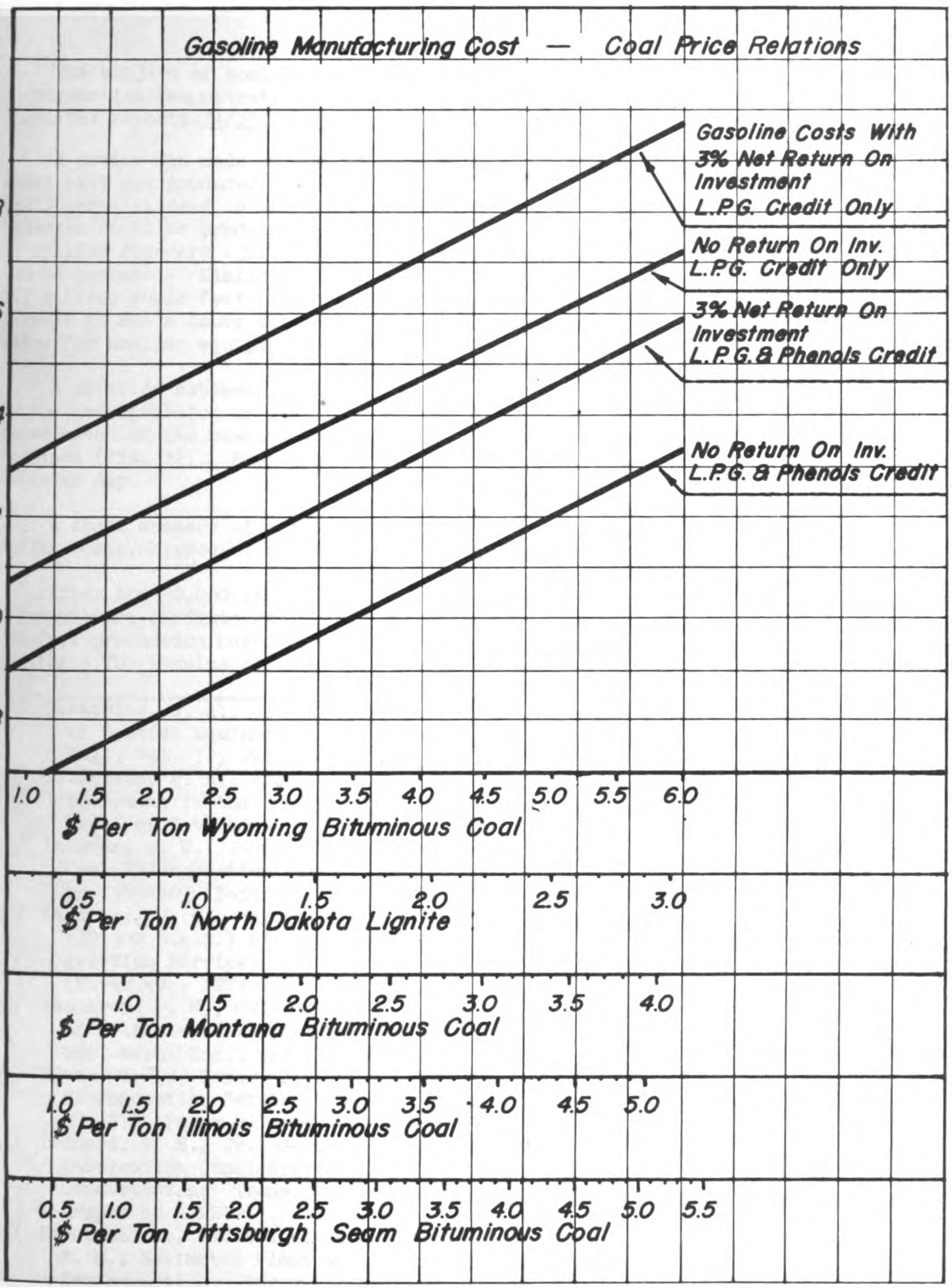

Figure 32. - Relation of gasoline manufacturing cost to coal prices for 30,000 bbl./day coal-hydrogenation plants.

The subject of coal hydrogenation and the special equipment used in the Coal-Hydrogenation Demonstration Plant at Louisiana, Mo., are described in a number of published reports.[31]/[32]/[33]/[34]/[35]/[36]/[37]/

A review was made of the possibilities of making hydrogen from retort gas and coker tail gas produced in shale-oil refining. It was found that both of these gases could serve as feed to the hydrocarbon-steam cracking operation, whereupon 98-percent hydrogen could be produced by shifting and CO_2 scrubbing of the effluent gases. From 21 million standard cubic feet of retort gas, 34.2 million cubic feet of hydrogen gas can be produced. Similarly, 3.25 million cubic feet of coker tail gas will produce 8.7 million cubic feet of hydrogen. Of the two gases, coker tail gas is preferred because it has a lower total sulfur content and a lower content of inerts, which makes for smaller equipment sizes.

A detailed estimate[38]/ was completed for the commercial production of gasoline by the hydrogenation of coal. Five coals typical of large deposits in widely separated areas of the country were selected, and the cost of commercial plants was determined (fig. 32). Each plant is to produce 30,000 barrels of liquid products per calendar day.

A short summary of coal consumption, products distribution, plant and manufacturing costs, byproduct credit, and net cost figures is given in table 18.

After the 30,000-barrel-per-day coal-hydrogenation plant estimate was completed, a comparative estimate was prepared investigating motor gasoline, avaition grade, and jet-fuel production based on Illinois No. 6 bituminous coal as raw material at $3.12 per ton. The results of this estimate are summarized in table 19.

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[31]/ Markovits, J. A., Coal Hydrogenation - Summary of Process and Brief Description of Special Equipment Used in Demonstration Plant at Louisiana, Mo.: Mech. Eng., vol. 71, July 1949, pp. 553-560.

[32]/ Markovits, J. A., Braun, K. C., Donovan, J. T., and Sandaker, J. H., Special Equipment in the Coal-Hydrogenation Demonstration Plant: Bureau of Mines Rept. of Investigations 4584, 1950, 40 pp.

[33]/ Donovan, J. T., Josenhans, M., and Markovits, J. A., High-Pressure Vessels in Coal-Hydrogenation Service: Trans. Am. Soc. Mech. Eng., vol. 72, May 1950, pp. 357-363 (Paper 49 - PET-6).

[34]/ Sandaker, J. H., Markovits, J. A., and Bredtschneider, K. B., High-Pressure (10,300 p.s.i.) Piping, Flanged Joints, Fittings, and Valves for Coal-Hydrogenation Service: Trans. Am. Soc. Mech. Eng., vol. 72, May 1950, pp. 365-372 (Paper 49 - PET-2).

[35]/ Laughrey, P. W., Gwillim, W. I., Schappert, H., and Markovits, J. A., Design of Preheaters and Heat Exchangers for Coal-Hydrogenation Plants: Trans. Am. Soc. Mech. Eng., vol. 72, May 1950, pp. 385-391 (Paper 49 - PET-1).

[36]/ Bruno, G. L., Geyer, F. W., and Markovits, J. A., Instrumentation for Coal-Hydrogenation Service: Trans. Am. Soc. Mech. Eng., vol. 72, May 1950, pp. 373-378 (Paper 49 - PET-4).

[37]/ Leonard, B. H., Jr., Gardner, G. D., and Markovits, J. A., Metallurgical and Fabrication Considerations in the Coal-Hydrogenation Demonstration-Plant Construction: Trans. Am. Soc. Mech. Eng., vol. 72, May 1950, pp. 379-383 (Paper 49 - PET-5).

[38]/ Hirst, L. L., Markovits, J. A., Skinner, L. C., Dougherty, R. W., and Donath, E. E., Estimated Plant and Operating Costs for Producing Gasoline by Coal Hydrogenation: Bureau of Mines Rept. of Investigations 4564, 1949 83 pp.

TABLE 18. - Estimate for commercial production of gasoline and byproducts by the hydrogenation of coal

	Bituminous coal			Subbituminous coal, Montana	Lignite, North Dakota
	Wyoming	Illinois	Pittsburgh seam		
Coal consumption and final products					
Consumption:					
Run-of-mine coal ...... tons/day	11,830	13,120	10,760	16,550	22,200
Production:					
Gasoline .... bbl./day	21,660	21,630	20,250	22,360	22,500
LP-gas .... bbl./day	7,100	7,130	8,510	6,400	6,260
Phenols .... bbl./day	1,240	1,240	1,240	1,240	1,240
Total, bbl./day	30,000	30,000	30,000	30,000	30,000
Plant-cost estimate[1]					
Hydrogenation section.. M dollars	$ 97,292	$ 99,625	$106,805	$ 98,635	$ 96,865
Gas-production section. do.	54,749	53,204	54,750	57,710	60,074
General and auxiliary plants .... do.	90,879	93,974	96,265	97,678	98 303
Total M dollars	$242,920	$246,803	$257,820	$254,023	$255,242
$1 bbl./day	8,097	8,227	8,594	8,467	8,508
Field labor .... M man-hours	21,736	22,013	22,803	22,733	22,968
Steel .... tons	158,000	159,000	167,000	164,000	165,000
Manufacturing costs					
Coal as mined .... $ /ton	$3.56	$3.12	$4.02	$1.10	$1.85
Personnel:					
Labor (operation)	2,400	2,440	2,540	2,510	2,520
Supervision and clerical	620	630	655	645	650
Total personnel	3,020	3,070	3,195	3,155	3,170
Costs, $ /day for 30,000 bbl./day of liquid products:					
Coal	$ 42,111	$ 40,925	$ 43,243	$ 18,162	$ 41,066
Other materials	17,156	17,443	19,286	18,212	18,103
Other direct costs	28,177	28,636	29,913	29,462	29,591
Indirect costs	17,847	18,136	18,945	18,662	18,746
Fixed costs (15 years amortization	55,212	56,072	58,591	57,386	57,967
Total	$160,503	$161,212	$169,978	$141,884	$165,473
Byproduct credit:					
Phenols at $0.10/lb.	$ 45,000	$ 45,000	$ 45,000	$ 45,000	$ 45,000
LP-gas at $0.08/gal.	23,856	23,957	28,594	21,504	21,034
Total credits	$ 68,856	$ 68,957	$ 73,594	$ 66,504	$ 66,034
Total net costs .... $ /day	$ 91,647	$ 92,255	$ 96,384	$ 75,380	$ 99,439
Unit costs ..... $ /gal. gasoline	$ 0.101	$ 0.102	$ 0.113	$ 0.080	$ 0.105

[1] Construction costs are based on the first quarter of 1948.

TABLE 19. - Total liquid-products estimate for 30,000-Barrel-per-day coal-hydrogenation plants

	Motor gasoline		Aviation gasoline		Jet fuel, JP-3	
	78 Octane No.	82 Octane No.	91 Octane No.	100 Octane No.	Motor-gasoline plant	Jet-fuel plant
Production of major product, bbl./day	21,630	21,630	20,121	11,920	24,260	24,260
Plant cost, M dollars	246,803	246,803	248,119	276,940	253,639	242,589
Cost of major product, gal., with credit for phenols	10.2	10.3	11.4	15.0	10.3	9.8
Without credit for phenols	14.2	14.3	15.7	17.9	14.0	13.5

One hundred octane-grade aviation gasoline produced by coal hydrogenation at a cost of 15 cents per gallon is competitive with that produced from crude oil. The production of this material is only 11,920 barrels per day in a 30,000-barrel-per-day plant. In addition, 12,208 barrels per day of 91 octane gasoline is made. If alkylate could be purchased, an additional 17,450 barrels per day of 100 octane aviation gasoline could be produced.

A study was made showing the effect of various percentages of return on capital investment for coal-hydrogenation plants producing various grades of gasoline. The results of this study are shown in table 20.

An estimate was made for converting the existing Morgantown Ordnance Works (600-ton-per-day synthetic ammonia plant) to a coal-hydrogenation plant, using Pittsburgh seam coal as feed. Based on the capacity of the existing hyper compressors of 70 million cubic feet of hydrogen per day, it was calculated that 7,600 barrels per calendar day of liquid-oil products could be produced at this plant. Coal requirement is 2,640 tons per day. Capital cost for additional new facilities is 47 million dollars. With coal at $4.50 a ton, the production cost of gasoline, after credit for LP-gas and phenols, is 17.3 cents per gallon. It is estimated that a personnel of 900 would be required to run the plant. If a hydroforming unit were installed, 2,216 barrels per calendar day of marketable aromatic chemicals could be produced.

An engineering and economic study was made to determine the cost of manufacture of a 900-B.t.u.-per-cubic-foot gas, using silt washings from anthracite coal-cleaning operations. The process selected was the gas-synthesis or Fischer-Tropsch process, using two fluidized-bed reactors. Two alternates for the gasification of the silt were selected: (a) Pressure dust gasification and (b) Lurgi gasification of pellets made from the silt. In each alternate the basic process units consist of a coal-preparation plant, gasification and oxygen plants, a dust-removal system, a water-gas shift for adjustment of hydrogen and carbon monoxide ratio, a water-washing step for removal of CO_2 and H_2S, a final sulfur-removal plant, and the synthesis step wherein hydrogen and carbon monoxide react over a suitable catalyst, producing water, which condenses out, and the finished city gas.

The cost of production, including no profit, but amortizing the total investment cost over a period of 15 years, is 54.5 cents per thousand cubic feet of 900-B.t.u.-per-cubic-foot gas, using the pressure dust gasification, and 63.5 cents, using the Lurgi gasification.

Capital investment cost for the two alternates is $57,955,000 and $59,950,000, respectively, for 65,650,000 standard cubic feet per calendar day of 900-B.t.u. city gas.

An engineering and cost comparison is shown in table 21.

TABLE 20. - Cost of producing various grades of liquid products by 30,000-barrel-per-day coal-hydrogenation plants

	Motor gasoline		Aviation gasoline		Jet fuel, JP-3	
	78 Octane No.	82 Octane No.	91 Octane No.	100 Octane No.	Motor gasoline plant	Jet-fuel plant
Products, bbl./day:						
Motor gasoline	23,000	23,000	-	-	-	-
Aviation gasoline, 91 O.N.	-	-	21,491	13,578	-	-
Aviation gasoline, 100 O.N. ...	-	-	-	11,920	-	-
Jet fuel, JP-3	-	-	-	-	25,670	25,670
LP-gas	7,130	7,130	8,639	2,582	4,465	4,465
Total	30,130	30,130	30,130	28,080	30,135	30,135
Plant cost, M dollars	246,803	246,803	248,119	276,940	253,639	242,589
Cost of major product, without credit for phenols:						
No return on investment[1]	14.2	14.3	15.7	17.9	14.0	13.5
40 percent income tax, 6 percent return[2]	21.6	21.7	23.7	33.0 [3](23.3)	20.8	20.0
3 percent return before income taxes, 40 percent income tax, 4 percent return after income tax[4]	17.8	17.9	19.6	25.1 [3](20.3)	17.3	16.6

[1] Includes cost of raw materials, operating costs, linear depreciation at 6-2/3 percent per year, a credit of other products at market value, no profit on investment and no income tax.

[2] Same as above but with 6 percent return after 40 percent income tax.

[3] If alkylate is purchased at 20 cents per gallon and blended with the surplus 91 octane to make all 100 octane (31,300 barrels) then the new selling price of the 100 octane is lowered to those figures shown in parentheses. Credit for the 91 octane is eliminated, but the capital charges are distributed over this larger production of 100 octane gasoline.

[4] Based on 50 percent of plant investment at 3 percent without taxes (loan or bond issue) and 50 percent of plant investment at 4 percent after 40 percent income tax (stock issue).

TABLE 21. - Engineering and cost comparison of pressure-dust and Lurgi methods of gasification

	Pressure-dust gasification	Lurgi gasification
Process coal .... tons/calendar day	5,760	4,194
Coal for power and steam .... do.	464	2,070
Coal for gasifier preheater .... do.	711	-
Coal for steam superheater .... do.	-	270
Total coal .... do.	6,935	6,534
Oxygen required .... do.	3,690	1,485
Operators per shift	43	59
Total plant investment including working capital and interest during construction	$60,155,000	$62,200,000
Total direct costs (silt coal at $12.5 and pellets at $2.50 per ton).	$ 19,194	$ 24,424
Total indirect costs	3,900	4,285
Total fixed costs	12,590	12,930
Total daily costs	$ 35,684	$ 41,639
Gas production, cu. ft./calendar day (900 B.t.u./cu.ft).	65,650,000	
Cost .... ¢/1,000 cu. ft.	54.5	63.5

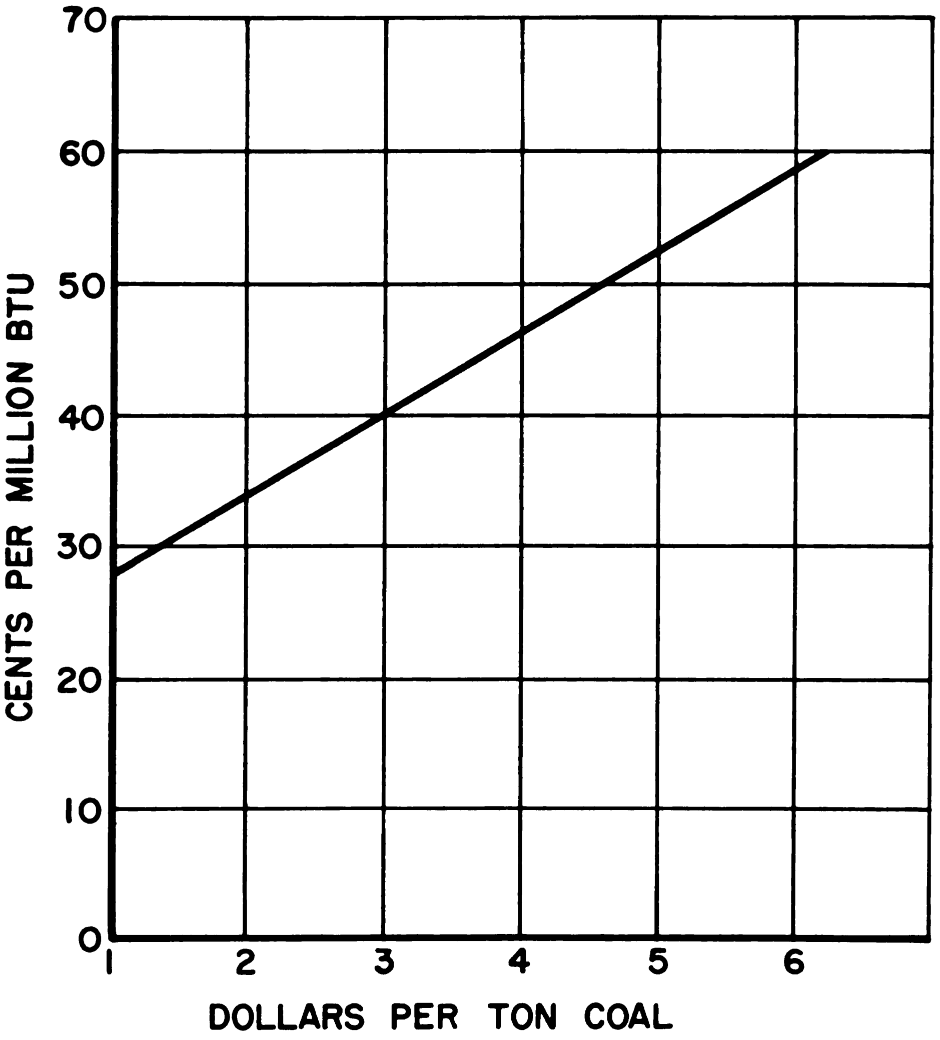

Figure 33. - Cost of byproduct city gas from synthetic liquid fuels plants vs. cost of coal at the plant site.

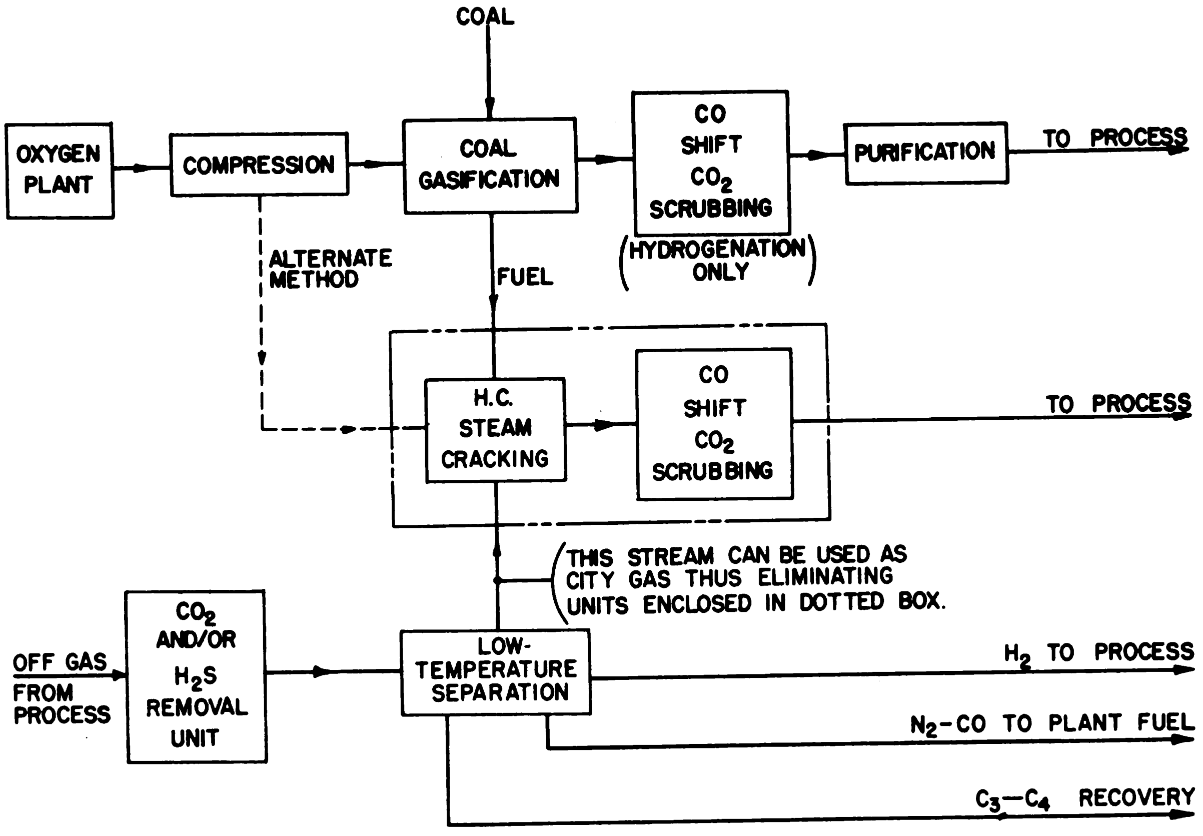

Figure 34. - Process units in synthetic liquid fuels plants making byproduct city gas.

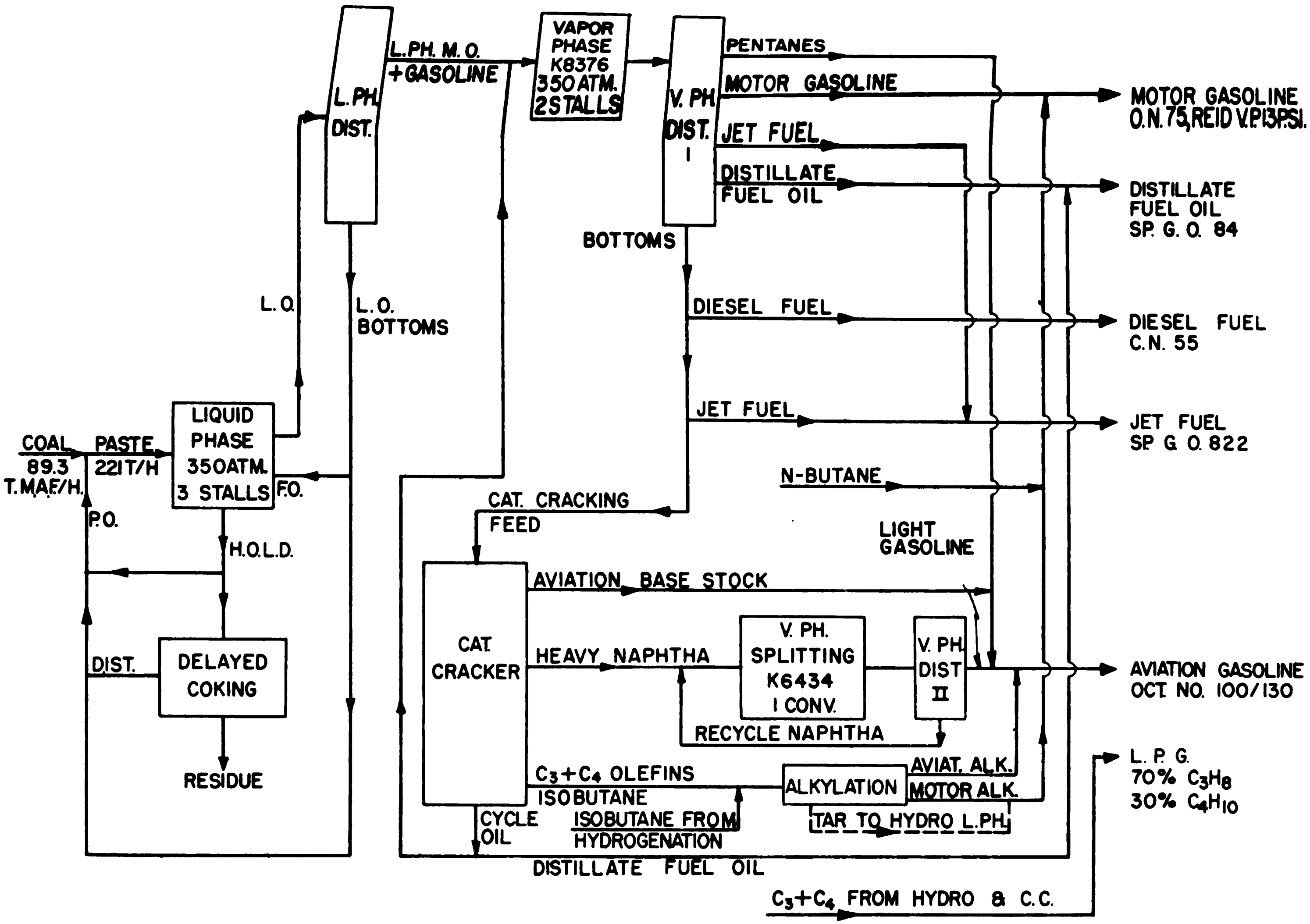

Figure 35. - Flow sheet for 10,000 bbl./day coal-to-oil plant using Alaskan subbituminous coal.

Data showing the possibilities of producing public-utility gas in a synthetic liquid fuel plant were discussed.[39/][40/]

Hydrocarbons recovered from the off gas of synthetic fuels plants have been suggested as a source of city gas. The value of this gas to the synthetic fuels process is obtained by determining the cost of additional coal-gasification facilities required to replace the hydrogen or synthesis gas normally made from the off gas. Its value has been calculated as 44 cents per million B.t.u., using a bituminous coal costing $3.56 per ton.

The use of this gas as a source of city gas will have an important and far-reaching effect upon the gas industry. Assuming 1 million barrels per day of synthetic liquid fuel (about 18 percent of present petroleum production) and only 75-percent utilization of byproduct hydrocarbons from synthetic liquid fuels plants, about 710 million cubic feet per day, or, roughly, 7-1/2 million therms per day will be available. This volume of gas is about 1-1/2 times the 1946 combined consumption of the cities of New York, Chicago, Philadelphia, and Detroit and about 90 percent of the total manufactured-gas production of the entire United States in 1944. Cost chart and flow diagram for this study are shown in figures 33 and 34.

Process flow sheet, material and heat balances, and cost studies were prepared for a 10,000-barrel-per-day synthetic fuels plant using Alaskan coal. It was proposed to use coal of the following analysis as the raw material:

		As mined	Moisture- and ash-free basis
H_2O		23.9	
Ash		5.1	
Volatile matter	percent		51.8
Fixed carbon	do.		48.2
H	do.		5.4
C	do.		71.5
N	do.		.9
O	do.		21.9
S	do.		.3
B.t.u./lb.			12,217

The proposed plant would yield the following products needed in Alaska:

	General specifications
Aviation gasoline	100/130 grade
Motor gasoline	75 A.S.T.M. O.N.
Diesel oil	Grade "C"
Distillate fuel oil and LP-gas	No. 2
Jet fuel	JP-3

The diversification of the liquid fuels produced shows the versatility of the coal-hydrogenation process when combined with a catalytic cracking unit, such as was contemplated for this plant. The flow diagram of the proposed plant is shown in figure 35.

[39/] Symonds, F. L., Laughrey, P. W., Skinner, L. C., Batchelder, H. R., and Donath, E. E., Potential New Source of Utility Gas Supply Offered by Synthesis Program - Impact of Synthetic Fuels: Am. Gas Assoc. Monthly, vol. 31, July-August 1949, pp. 18-20, 24.

[40/] Symonds, F. L., Laughrey, P. W., Skinner, L. C., Batchelder, H. R., and Donath, E. E., Public Utility Gas as a Byproduct of Synthetic Liquid Fuels Production: Proc. Am. Gas Assoc., 1949, pp. 789-795.

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A summary of a cost study on the coal-to-oil plant in Alaska compared to a similar plant in the United States is given below:

		Costs, plant located in U. S.	Costs, plant located in Alaska
Coal at plant site	$ /ton	1.85	6.50
Labor	$/hour	1.75	2.75
Power	c/kw.-hr.	0.5	0.5
Period of amortization	years	15	15
Total product cost	c/gallon	15.0	35.0
Total capital cost		$93,700,000.00	$243,800,000.00

Technical Reports and Foreign Document Work

Abstracts and Bibliographies

Compilation of synthetic liquid fuels abstracts has been continued in the bimonthly form, and approximately 1,000 copies of each issue are supplied to a mailing list. A comprehensive coverage of current literature and patents is being maintained.

A bibliography of pressure hydrogenation was completed, which contains three sections: 2,503 annotated literature references; 3,569 annotated patent references; and a subject literature index, a subject patent index, and a numerical patent index. Preparation of a bibliography of the Fischer-Tropsch process, was begun. Completion of this compilation has been delayed somewhat beyond the time originally planned in order that several hundred German patent applications could be searched and those of interest incorporated in the bibliography. A bibliography of Bureau of Mines investigations on the production of synthetic liquid fuels was completed.[41]

A general review of the research activities currently under way in the Bruceton laboratories was published.[42]

Foreign Documents

Indexing of foreign documents was continued. About 1,000 additional documents were reviewed and indexed during the fiscal year. These include the separate documents in 25 FIAT reels which were later transposed to TOM reels 281-305. All documents in these files, except those in TOM reels 1-279, of special interest to the synthetic liquid fuels program are now subject indexed.

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[41] Golumbic, Norma, Anderson, Hazel C., and Grass, Robert C., Revised Bibliography of Bureau of Mines Investigations on the Production of Liquid Fuels from Oil Shale, Coal, Lignite, and Natural Gas (to 1949): Bureau of Mines Inf. Circ. 7534, 1949, 53 pp. (Revision of Inf. Circ. 7304 by Arno C. Fieldner and Paul L. Fisher.)

[42] Grass, R. C., and Storch, H. H., Coal-to-Oil Research at Bruceton, Pa.: Chem. Eng. News, vol. 28, Feb. 27, 1950, pp. 646-648.